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DEVELOPMENT OF FINE DIAMETER HIGH-PURITY WIRE FROM ZONE-REFINED BERYLLIUM

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Final Report
December, 1962

Contract NOw 62-0067-c

RESEARCH AND DEVELOPMENT

The Brush Beryllium Co.

CLEVELAND, OHIO

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ABSTRACT

Zone-refined crystals of both thermally and electrolytically reduced beryllium were converted to polycrystalline aggregates by a combination of deformation and heat treatment. The resulting polycrystalline aggregates were subjected to deformation at 450° C by standard beryllium wire-drawing techniques. A limited amount of metallurgical evaluation of the resulting wire was accomplished.

It was found that combination of twinning and primary recrystallization was required to convert the single crystal to a truly polycrystalline aggregate.

The wire produced from zone-refined beryllium exhibited usable structural tensile-properties at room temperature.

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I INTRODUCTION

The U.S. Navy, Bureau of Weapons, has been sponsoring beryllium purification studies at The Franklin Institute, Philadelphia, Pennsylvania, under a series of contracts. These studies have been quite successful and have produced, by multiple-pass zone refining procedures, purified metal which as a single crystal behaves in a very ductile manner.

It was then of interest to gain some information about a polycrystalline aggregate of this zone-refined beryllium in order to probe the structural potential of this material. Wire was chosen as the most advantageous mill form for this probe. Beryllium wire of commercial, block-pressed purity has been hard-drawn to exceptionally high strength. [1] Wire provides a relatively inexpensive means for the determination of ductility under conditions of triaxial strain (longitudinal, radial, and circumferential strain so long as the wire retains its cylindricity during test deformation).

This program was designed to convert zone-refined beryllium single crystals to polycrystalline aggregates through deformation and heat treatment. The polycrystalline aggregates were to be deformed subsequently by wire drawing in a manner which would produce maximum strengthening.

The primary objective of this program was to fabricate wire at a fine diameter from zone-refined beryllium without chemical contamination. Of subordinate interest in this program was an evaluation of the metallurgical properties of this wire.

This report describes the manner in which the primary objective was accomplished. In the course of accomplishing this primary objective, a number of metallurgical measurements were made in order to guide the fabrication procedures. These measurements are described also in this report. Their inclusion is principally as a "contribution to the data" since these measurements were not made in the systematic manner which is required for valid conclusions.

II EXPERIMENTAL PROCEDURE

A. Characterization of Single Crystals

The input material for this program consisted of single crystals of beryllium which were produced by zone-refining techniques.

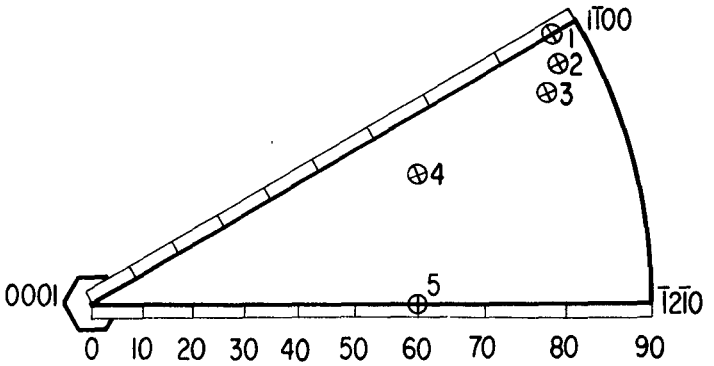
The first three crystals were produced by The Franklin Institute from electrolytically reduced beryllium. These crystals (identified as A, B, and C) were produced from hot-extruded Pechiney "SR" flake by passing each crystal with three zones. The first zone was passed at 2 inches per hour; the last two zones were passed at 1/2 inch per hour. (2) These crystals were 0.190 to 0.230-inch diameter by 7 to 7 1/4-inches long and appeared to have an excellent surface finish.

The fourth crystal was produced by The Brush Beryllium Company from thermally reduced beryllium. This crystal (identified as 31) was produced from cast "selected pebble" by passing the crystal with six zones. This crystal was about 0.350-inch diameter by 6 3/4-inches long.

The fifth crystal was produced by The Franklin Institute from thermally reduced beryllium. This crystal (identified as D) was produced from cast "selected pebble" by passing the crystal with seven zones. This crystal was about 0.980-inch diameter by about 6-inches long. Only the first 2 inches (from start of zone) were used in this program.

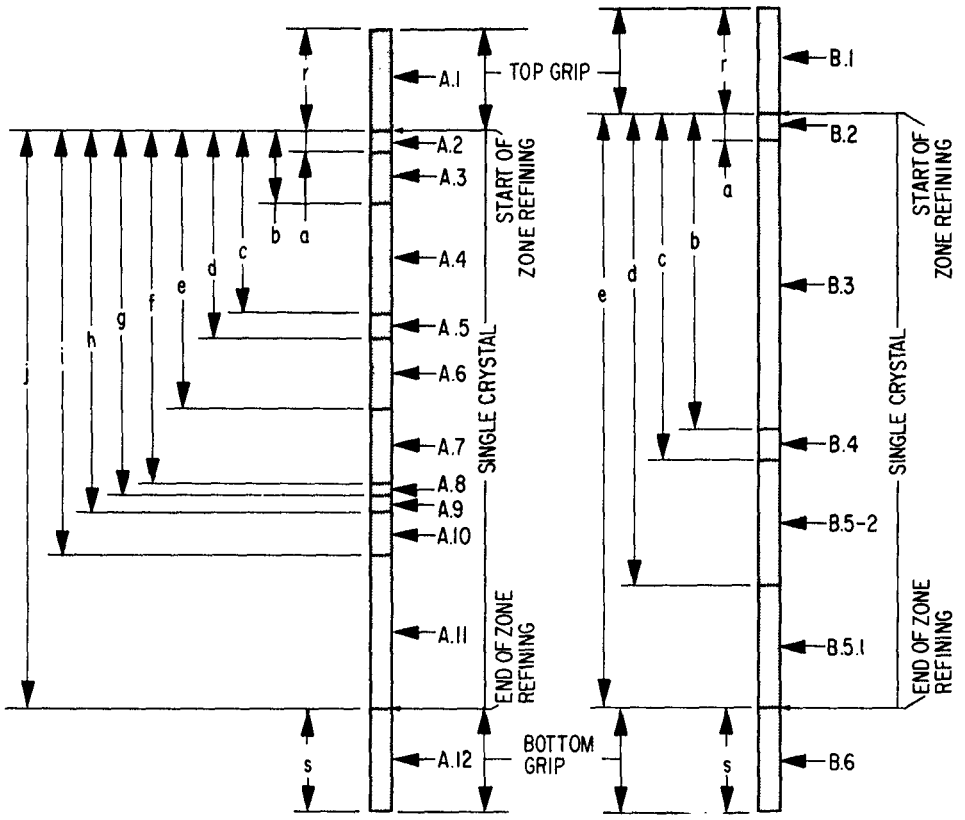
The angular relationship between the rod axis and the crystal lattice is given for each crystal in the unit stereographic triangle in Figure 1.

These crystals were subdivided to provide blanks for metallography, chemical analysis, and fabrication. Crystals A, B, C, and 31 were cut with a radiac which was equipped with a dashpot-feed-regulation-control. Crystal D was cut by spark-discharge at The Franklin Institute. The subdivisions were assigned unique identities as is indicated for crystals A and B in Figure 2. Only one section from crystal C was used and this blank was located in the last 1 1/2 inches of single crystal (similar to B.5-1 in Figure 2). Crystal 31 was cut much as was crystal B in that samples for chemical analysis were cut at positions corresponding to B.2 and B.4 in Figure 2 while the length corresponding to B.3 was cut in half and the two blanks were identified as 31 B (top) and 31 C. Crystal D was sampled twice to produce two blanks, each of which was approximately a 1-inch long right cylinder. The first blank came from the first inch from start-of-zone refining and was identified as D.1. This blank was not exactly a single crystal but contained at least two easily discerned grains



LEGEND:
1. Rod Axis for Franklin Crystal A
2. Rod Axis for Franklin Crystal B
3. Rod Axis for Franklin Crystal C
4. Rod Axis for Brush Crystal 31
5. Rod Axis for Franklin Crystal D

Fig. 1 - Orientation of Rod Axes of Zone-Refined Single Crystals of Beryllium



CRYSTAL A *CRYSTAL B*

SYMBOL	DIMENSION (INCH)
r	$1\frac{1}{4}$
a	$\frac{3}{8}$
b	$\frac{11}{16}$
c	2
d	$2\frac{1}{4}$
e	$3\frac{1}{4}$
f	$4\frac{1}{8}$
g	$4\frac{5}{16}$
h	$4\frac{1}{2}$
i	$5\frac{1}{16}$
j	7
s	$1\frac{3}{16}$

SYMBOL	DIMENSION (INCH)
r	$1\frac{1}{4}$
a	$\frac{3}{8}$
b	$3\frac{7}{8}$
c	$4\frac{1}{4}$
d	$5\frac{3}{4}$
e	$7\frac{1}{4}$
s	$1\frac{1}{4}$

Fig. 2 - The Subdivision of Crystals A and B

near the surface at one end. The second blank, identified as D.2, came from the second inch of the crystal and appeared to be a single crystal.

Samples of crystals A and B were examined metallographically in transverse section. The purpose of such examination was to evaluate the cleanliness of these crystals and to look for the presence of subgrains. Since the optical plane was normal to the cylindrical (rod) axis of each crystal, a $\{10\bar{1}0\}$ plane was within 4 degrees of coincidence with the optical plane (see Figure 1). This fact made metallographic examination with polarized light a very powerful tool for detecting misregistry of basal planes[3]; that is, grain boundaries which are not just spins about a $\langle 0001 \rangle$ which is common to both sides of the boundary. A Bausch and Lomb Research Metallograph was used for these examinations and the results are given in Table I. These observations turned out to be only partially representative of the various areas of these crystals in that a copious amount of non-metallics were found in crystal B at a later stage in the processing, as will be discussed later.

Samples from crystals A, B, and 31 were subjected to chemical analysis by emission spectrography. The results of these determinations are presented in Table II.

The residual resistance ratio for specimens A.4 and A.7 was determined by the eddy current decay method. These determinations were made by Mr. R. Genberg, Physics Department, Case Institute of Technology. Basically, this is the ratio of the resistivity at 20°C to the resistivity at -269°C. The residual resistance ratio for A.4 was 18.0 and for A.7 was 17.3. This is considered to be about "par" for this material.[4]

Single crystal mechanical property data were not available for crystals 31 and D. Very little information was available for crystals A, B, and C; the room temperature critical resolved shear stress for basal glide was found to be about 600 psi for a "sister" crystal to these three.[4]

However, if some results may be anticipated at this point, a measure of the resistance (at 450°C) to swaging deformation was available for crystals A, 31, and D.1. In addition, unpublished work which was sponsored by The Brush Beryllium Company produced the same information for a 6-pass zone-leveled crystal which was made from the same starting material and to the same crystal size as was crystal 31. This zone-leveled crystal, crystal 35, was determined to be unrefined, by-the-large, in that the concentration of each metallic element (except titanium) was not altered significantly by zone leveling.

TABLE I

METALLOGRAPHIC EXAMINATION OF TRANSVERSE SECTIONS OF CRYSTALS A AND B; 100X, PD

Sample Identity	Disposition	
	Grain Boundaries [a]	Non-metallic Inclusions [b]
A. 3	None	None
A. 5	None	One located close to surface; dimensions were 0. 0025-inch by 0. 025-inch
A. 8	None	One located close to surface; dimensions were 0. 0009-inch by 0. 04-inch
A. 10	None	Numerous small, acicular indications which formed a band along a diameter of the section; dimensions of individual particles averaged 0. 0006-inch by 0. 005-inch
B. 2	None	None
B. 5	None	None

[a] A misregistry of 5 degrees or greater is required for detection of a boundary.

[b] No apparent orientation habit with basal planes.

TABLE II

CHEMICAL ANALYSIS OF VARIOUS SAMPLES OF PURE BERYLLIUM

Element	Analysis (ppm)[a] and date of analysis of: A. 12[b] 4/27/62	A. 2 3/22/62	A. 3 4/27/62	A. 5 4/27/62	A. 8 4/27/62	A. 9 3/22/62	B. 2 4/27/62	B. 4 4/27/62	Top of[c] 31 B 6/20/62	Bottom of[c] 31 C 6/20/62
C	920	--	--	--	--	--	--	--	--	--
Al	15	55	12	10	15	40	15	12	-25	-25
Cr	25	8	8	8	30	-8[c]	30	20	-6	-6
Fe	85	140	70	85	100	125	70	100	100	270
Mg	-25	50	-25	-25	-25	35	-25	-25	-25	-25
Mn	10	6	-6	-6	6	15	-6	6	-6	-6
Ni	70	120	90	60	70	90	120	70	70	70
Ti	-6	--	-6	-6	-6	--	-6	-6	-6	-6
Cl	--	--	--	--	--	--	--	--	--	--
Ca	-85	-85	-85	-85	-85	-85	-85	-85	-85	-85
Co	-3	-1	-3	-3	-3	-1	-3	-3	-3	-3
Cu	10	55	20	25	90	30	20	10	50	50
Si	70	100	70	55	85	55	70	55	30	30
Zn	-55	-55	-55	-55	-55	-55	-55	-55	-55	-55

[a] All samples converted to BeO; results are on a Be metal basis. Emission Spectrography

[b] Hot-extruded Pechiney "SR" flake; not zone refined.

[c] Minus sign indicates "less than".

The measure of resistance to deformation is formed by comparing the actual reduction ratio sustained by the beryllium with the reduction ratio applied to the jacketed beryllium assembly. The applied reduction ratio is defined by the reduction ratio per swaging cycle and the number of cycles applied. (See Part B of this Section for a more complete description of the swaging procedure.) The comparison is formed by dividing the "actual" by the "applied".

The data reduce to show the following measures of resistance to deformation at 450° C:

Empty Steel Jacket; I. D.	> 50 (for 3 or more swaging cycles)
Specimen A. 4	0. 648
Specimen A. 6	0. 745
Specimen A. 11	0. 701
Specimen 31 A	2. 175
Specimen 31 B	2. 175
Specimen D. 1	1. 430
Specimen 35	0. 310

It should be noted that samples 31 A, 31 B, D. 1, and 35 all had initial orientations which were significantly different from the initial orientation of crystal A. It is believed that this difference could have caused these four specimens to "run-out" somewhat faster than crystal A during the early stages of deformation. This difference in "run-out" rate should have decayed completely by the time that polycrystallinity was achieved, perhaps by the end of the second swaging cycle. Thus, the above data are but a first approximation of questionable precision. None-theless, these data do seem to indicate that crystals 31 and D had effective flow stresses at 450° C which were equal to or less than that of crystal A. As expected, crystal 35 showed the highest resistance to deformation. The value for the empty steel jacket indicates the value that would be obtained for beryllium which possessed an effective flow stress of zero at 450° C. On the other end of the scale, beryllium with an effective flow stress of infinity at 450° C would produce a value of zero for the above index.

B. Fabrication Procedures

Blanks were cut from the crystals and were jacketed in mild-steel, seamless tubing. The tubing dimensions were chosen to accommodate

both crystal diameter and the die schedule. A wall thickness to OD ratio of 10 was maintained for all crystals. The steel was degreased and the crystal was slipped inside. The jacketed beryllium and the required dies were heated to 450° C in an electric furnace (ambient atmosphere). The compact was then subjected to a series of swaging, or in the case of crystal C rod drawing, passes (about 10% reduction in area per pass) until the OD of the assembly was reduced to original ID of the jacket. This constituted one "cycle". Anneals were applied after passing each die during the first cycle. The assembly was then placed in a second piece of tubing and subjected to a second deformation cycle. Note that the first jacket was left in place and now there were two pieces of steel on the beryllium. The second cycle consisted of the same dies as were used previously and an anneal. This cycling was continued until the beryllium core had reached the desired dimensions and was covered with as many separate jackets as there were cycles. (The dimensions of the beryllium were tracked by radiographs.) At this point the steel was removed in dilute nitric acid.

The resulting polycrystalline aggregates were then reduced in diameter by standard beryllium wire-drawing techniques.[1] A 10% reduction in area equidrafting die schedule was employed at 450° C.

C. Metallurgical Measurements

Metallographic examinations were performed using procedures standard for beryllium.[5] A Bausch and Lomb Research Metallograph equipped with polarized light was used for these examinations.

Preferred orientation in polycrystalline aggregates was determined using normal incidence collimated copper K-alpha radiation (30 KV and 18 MA) and a forward reflection pin-hole camera. The diffracted beams were recorded on Kodak No-Screen film. A transmission photodensitometer was used to measure the photodensity of the recordings. The values of photodensity were reduced to diffracted intensity by standard calculations.[6]

Tensile testing was performed using an Instron Model TT-C-L machine.

III RESULTS AND DISCUSSION

A. Conversion of Single Crystals to Polycrystalline Aggregates

Four blanks from crystal A were swaged successfully to polycrystalline aggregates. As might be expected, the diameter of the beryllium was not very uniform and the beryllium was somewhat "out of round" in local regions along these lengths.

The approximate dimensions of these swagings upon dejacketing and the number of cycles to which they were subjected are:

A. 4	6 cycles	0.056 $\pm$ 0.010-inch diameter by 24-inches long.
A. 6	6 cycles	0.060 $\pm$ 0.010-inch diameter by 12 3/4-inches long.
A. 10	5 cycles	0.080 $\pm$ 0.020-inch diameter by 3 1/2-inches long.
A. 11	7 cycles	0.072 $\pm$ 0.010-inch diameter by 18-inches long.

In all of the preceding cases the jacketed beryllium was annealed at 815° C for 1 hour and air cooled after each of the first six swaging passes. With the exception of sample A. 6, the jacketed beryllium was annealed also at 815° C for 1 hour and air cooled after each cycle. Sample A. 6 was treated identically until the fourth swaging cycle. After cycles 4, 5, and 6 sample A. 6 was annealed at 730° C for 30 minutes and air cooled.

Three blanks from crystal B were subjected to swaging. The approximate dimensions of these swagings upon dejacketing and the number of cycles to which they were subjected are:

B. 3-1	6 cycles	0.073 $\pm$ 0.010-inch diameter by 14 3/4-inches long.
B. 5-1	4 cycles	0.070 by 0.150-inch elliptical by 3 pieces (2 1/2-inches, 1 3/4-inches, and 1-inch long).
B. 5-2	7 cycles	0.033 by 0.120-inch elliptical by 21-inches long.

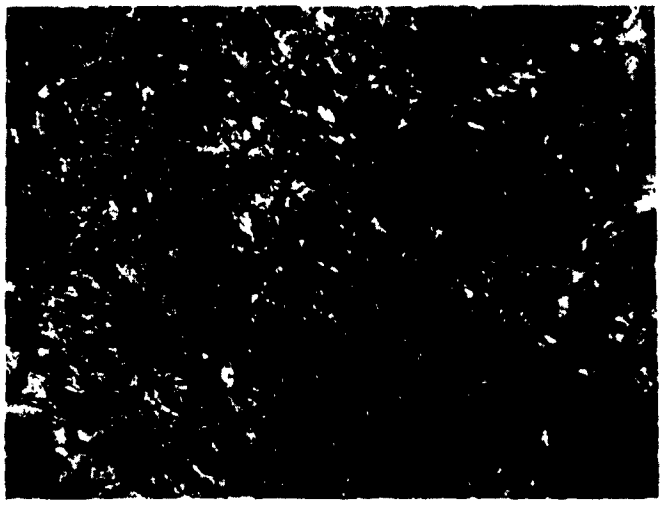
Sample B. 3-1 was processed in a manner which was identical with that described for sample A. 6. Samples B. 5-1 and B. 5-2 were annealed in their jackets at 730° C for 20 minutes and air cooled after each of the first six swaging passes, as well as after each swaging cycle. An exception to the preceeding sentence was the application of an anneal at 815° C for 60 minutes and air cool to sample B. 5-2 after swaging cycle number 6.

One blank from crystal C was jacketed and subjected to deformation by rod drawing. The schedule of drawing dies was the same as the schedule of swaging dies which was used on crystals A and B. The annealing practice was identical with that which was used for samples A. 4, A. 10, A. 11, and B. 3-1. This sample, C. 6, was dejacketed after 7 cycles during which the radiographs indicated more and more ellipticity and was found to be elliptical much as in the cases of B. 5-1 and B. 5-2. The beryllium dimensions were approximately 0. 080 $\pm$ 0. 010 by 0. 046 $\pm$ 0. 002 (elliptical) by 17-inches long.

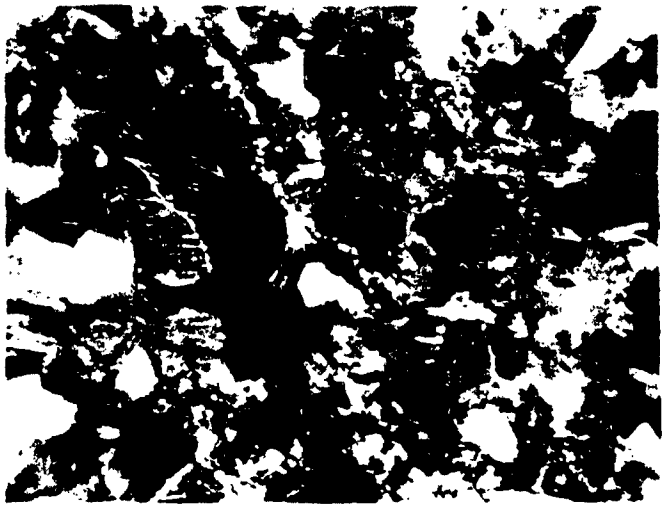
The first metallurgical measurement to be made on these swaged wires was metallographic examination in transverse section. Sections from as-swaged wires exhibited microstructures which were populated profusely with prominent deformation features such as bent basal planes, twins and/or deformation bands (kinking, segmentation). Examples of this are presented in Figure 3 A and in Figure 4. As will be shown below, the acicular deformation features are most probably twins. These features made almost meaningless any estimation of the wrought transverse grain size.

Upon annealing these swaged structures at 815° C for 30 minutes, the deformation features disappeared and an equiaxed structure such as is shown in Figure 3 B was observed. Of primary importance in Figure 3 is the network of "stringers" in this microstructure. These stringers were identified as non-metallic inclusions during metallographic examination and, as will be shown in Part B of this section, are most probably beryllium oxide. It is believed that this BeO was present in the "as-received" single crystals. It seems most improbable that these inclusions were introduced by chemical contamination during swaging since the beryllium was protected by the steel jackets. Further, the non-metallic inclusions reported in Table I had the same optical characteristics as those in Figure 3.

The results of the metallographic examination of sample B. 5-1 are most interesting. It should be recalled at this point that this sample was never subjected to temperatures above 730° C during processing. Further, radiographs showed that this sample became more and more elliptical as



A

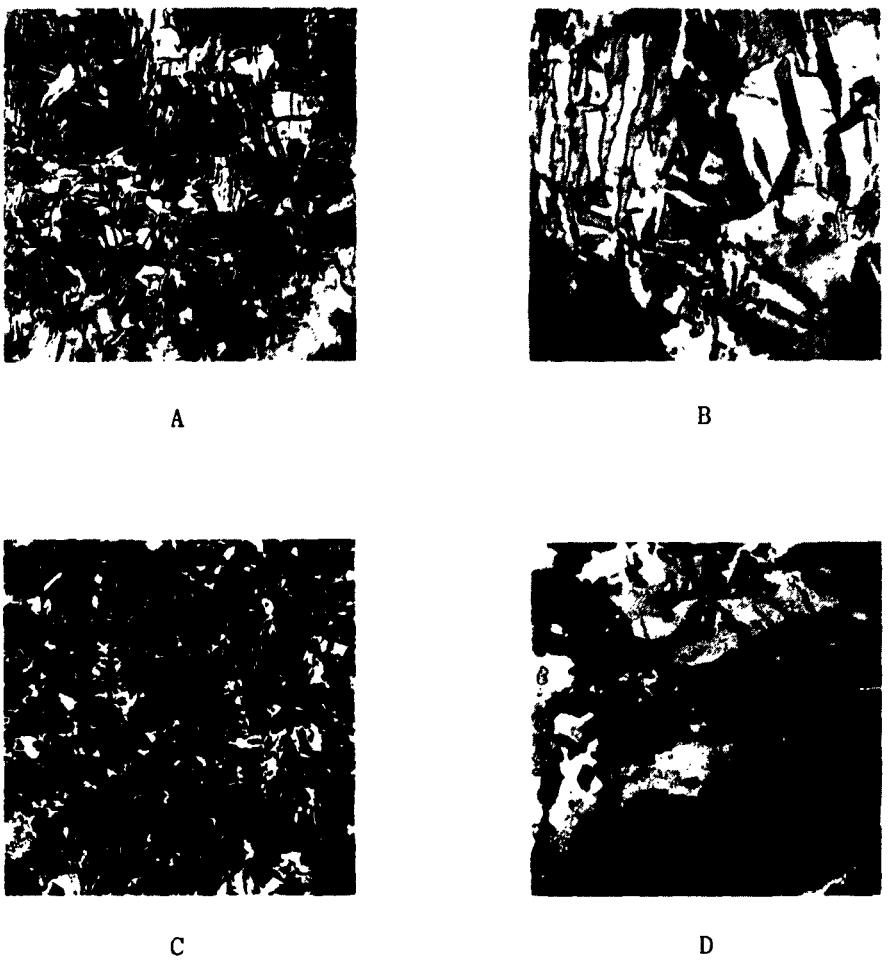


B

Fig. 3 - Photomicrographs of Sample B. 3-1
after Swaging

A. Transverse Section; As-Swaged; 100X, PD.

B. Transverse Section; Swaged and Annealed at 815°C for 30 Minutes-A. C.; 100X, PD



A B

C D

Fig. 4 - Photomicrographs of Sample B. 5-2; As-Swaged

A. One End of B. 5-2; 100X, PD

B. Same as (A); 750X, PD

C. The Opposite End of B. 5-2; 100X, PD

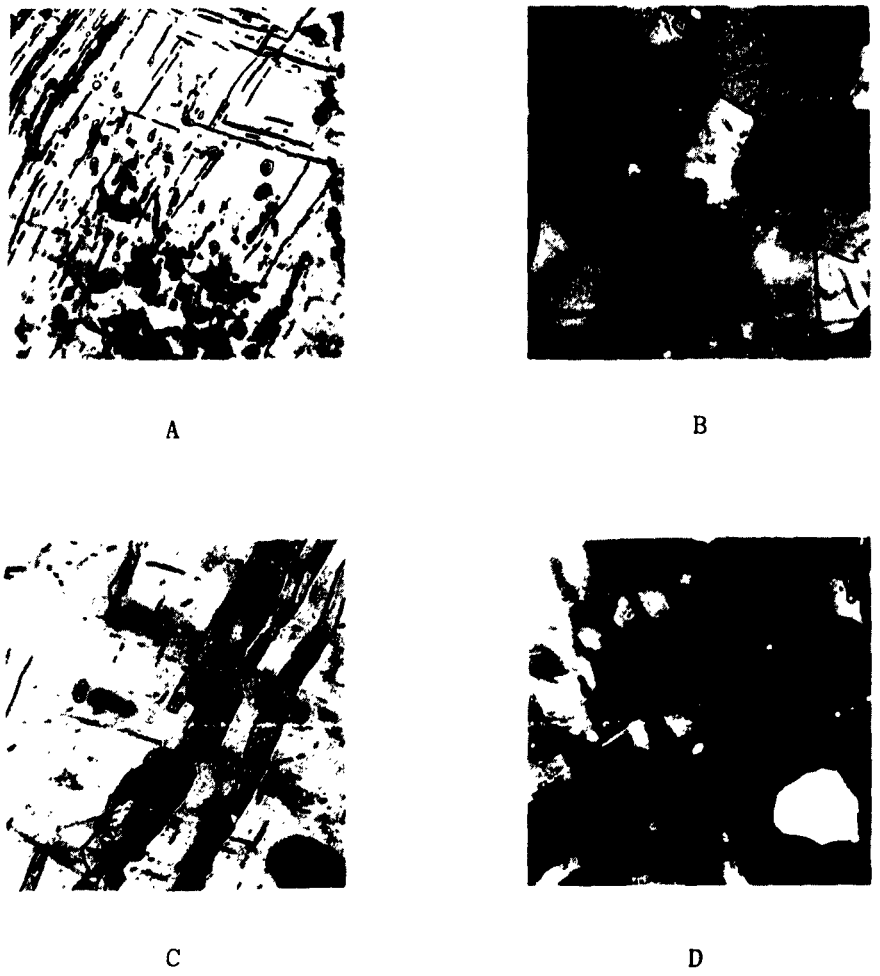
D. Same as (C); 750X, PD

processing continued. This sample fractured in two places sometime during the processing. As near as could be determined, the fracture planes were approximately parallel to a $\{\bar{1}2\bar{1}0\}$ plane in the original crystal. The major axis of the elliptical transverse section was approximately coplanar with the rod axis and the c-axis of the original crystal.

Figure 5 presents some photomicrographs from sample B. 5-1. The optical plane corresponds very closely to a $\{10\bar{1}0\}$ plane of the original crystal. So long as pronounced polycrystallinity has not been attained, this correspondence should not have been altered by the swaging deformation. The structures in Figure 5 serve to illustrate that the matrix of sample B. 5-1 was polygonized into subgrains but might still be described, by the large, as an imperfect single crystal. This allows an almost positive identification of the acicular deformation features as being twins. Figure 6 presents a $(10\bar{1}0)$ projection of a beryllium single crystal. Also included are the traces in the $(10\bar{1}0)$ of the twin composition planes, $\{10\bar{1}2\}$, of the three possible twin orientations. These traces are shown as dashed lines. These dashed lines delineate also the major axis of acicularly shaped twins. Note that the angle of intersection of traces 1 and 2 in Figure 6 is about equal to the intersection angle of the acicular deformation features in Figure 5 A. The strain-accommodation criterion of Schmid and Boas, as adapted for beryllium by Lee and Brick, [7] provides a rational explanation for the formation of these twins during swaging (by compression along $\langle\bar{1}2\bar{1}0\rangle$).

Another important point here is that the ellipticity of sample B. 5-1 suggest that there was little or no deformation assignable to slip directions which have components parallel to the c-axis. If this be so, then this high-purity beryllium probably deforms at 450° C by the same mechanisms and along the same slip systems as does commercially-pure beryllium. These arguments are not necessarily in conflict with the fact that swagings from crystal A did not become elliptical. The higher annealing temperature which was used in all of the early stages of deformation of crystal A is believed to have induced rather complete recrystallization prior to the development of a serious degree of ellipticity. Such extensive recrystallization of a profusely twinned structure could promote a polycrystalline aggregate which had a rotationally symmetrical (about the rod axis) response to the swaging stresses. The formation of such an aggregate would require that some of the twins grow (or survive) to become recrystallized grains.

This leads into the apparently anomolous case of sample C. 6 which became more and more elliptical with increasing deformation, despite the employment of the 815° C annealing temperature during the early stages



A B

C D

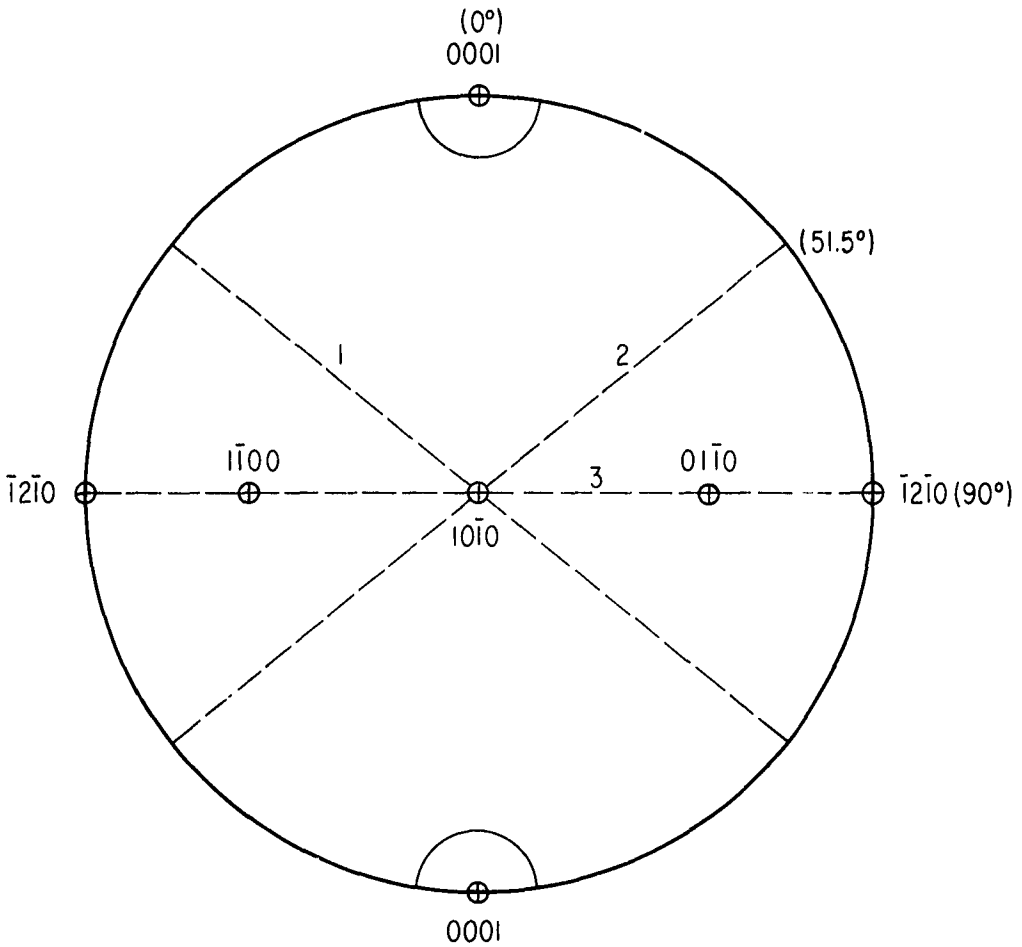
Fig. 5 - Photomicrographs of Sample B.5-1; As-Swaged and Annealed (730°C).

A. 100X, PD

B. Recrystallized Region. 750X, PD

C. Twinned Region; Stage at Zero-Degree Setting. 750X, PD

D. Same as (C); Stage at 45-Degree Setting. 750X, PD



Notes: 1. Trace of $(01\bar{1}2)$ Twin Composition Plane in $(10\bar{1}0)$.
2. " " $(1\bar{1}02)$ " " " " (").
3. " " $(10\bar{1}2)$ " " " " (").

Fig. 6 - $(10\bar{1}0)$ Projection for Beryllium With Traces of Twin Composition Planes. c/a = 1.566

of deformation. It should be recalled that this sample was deformed by passing the jacketed crystal through rod-drawing dies at 450° C. This technique applies a somewhat different stress state to the jacketed beryllium than does swaging. The swaging dies were "relieved" heavily at the parting plane and thus did not apply a very serious amount of radial compression (on any given stroke) to two of the four quadrants; the longitudinal stress is mildly compressive while the die is deforming the workpiece. Rod-drawing dies, on the other hand, are not relieved in two quadrants and thus exert potentially a completely rotationally symmetrical compressive radial stress; the longitudinal stress is significantly tensile.

The important point here is that during rod-drawing of sample C. 6, the compressive radial stress applied by the die along a $[\bar{1}2\bar{1}0]$ direction in the beryllium was accompanied coincidentally by a compressive radial stress applied by the die along a $[0001]$ direction in the beryllium. This was not the same case as in swaging where the stress along a $[0001]$ was very small or absent due to the relief of the dies at their parting plane.

Now when a beryllium crystal twins under the influence of compression along a $[\bar{1}2\bar{1}0]$ direction (that is, contracts in this direction to relieve the compressive stress), the crystal must extend along a $[0001]$ as a consequence of the geometry of twin formation. Extension parallel to the c-axis isn't of much importance in the twinning process unless there is a significant compressive stress being applied along the c-axis. Such a compressive stress was existent during the rod-drawing of sample C. 6 and formed what might be described as a "potential-energy-barrier" (elongation in the face of a compressive stress) to twin formation. This argument holds for all three twin systems. There is no need to invoke the existence of the longitudinal tensile stress along the rod axis during drawing even though this stress, by itself, forms a potential-energy-barrier to twin formation on system number 3 in Figure 6.

Thus, it would appear from the foregoing arguments that twinning and extensive recrystallization must both be active mechanisms in order to convert successfully the single crystal into the desired polycrystalline aggregate. Lacking one or the other of these mechanisms during the early stages of deformation leads to the development of a highly defective single crystal rather than the development of true polycrystallinity.

The preferred orientation of the swaged wire was probed also. A forward reflection pin-hole camera X-ray diffraction pattern was produced from a piece of sample A. 11 in the swaged and annealed (815° C) condition (after seven swaging cycles).

This pattern was analysed for photodensity. Readings on the $\{0002\}$ Debye ring were taken at each 2-degree interval of the angle between the $[0001]$ and a plane normal to the wire axis. Readings were taken also of the background immediately inside the $\{0002\}$ Debye ring. The photodensity values were reduced to intensities, (6) and the ratio of the line intensity to the background intensity was formed for each angular position. These values are presented graphically in Figure 7. Note that basal planes are parallel to the wire axis at the zero-degree position, and that no information on the $\{0002\}$ distribution is available in the angular intervals of -90 to -64.5 degrees and +64.5 to +90 degrees. Also included in Figure 7 are values of $\{10\bar{1}0\}$ reflection intensity. The primary significance of these values is that the value of diffracted intensity at the 30, 90, and 150-degree positions would be zero when all basal planes are parallel to the wire axis and all $[10\bar{1}0]$ are parallel to the wire axis; the values at the 60 and 120-degree positions would be at some finite maximum in this instance. Noted in Figure 7 is a value of "DPT" which is used herein as an abbreviation for "degree of perfection of $\langle 10\bar{1}0\rangle$ texture". The DPT parameter has the property of being unity for randomly oriented prism planes and of being infinity for a perfect $\langle 10\bar{1}0\rangle$ fiber axis.

Harking back to the prismatic information in Figure 7, it may be seen that the diffracted intensity is highest at the 30 and 150-degree positions. One would expect the values at the 30, 90, and 150-degree positions to be approximately equal and certainly lower than the values at the 60 and 120-degree positions. This expectation assumes that the prismatic alignment is accomplished by spins about individual $\langle 0001\rangle$ when basal planes are nearly parallel to the wire axis. The prismatic alignment in this specimen does not have such a characteristic, and thus suggests that a significant amount of material is oriented with basal planes well out of parallel with the wire axis, specifically in the angular range of 20 to 40 degrees. The basal atomic-population-distribution curve verifies this interpretation. Note that basal plane tilts within the 20 to 40-degree range will not affect the DPT value very much, if at all, because such tilts do not significantly affect the diffracted intensity at the 90-degree position and affect the diffracted intensity at the 60-degree position but slightly. By the same token, it may be reasoned that very little material is oriented with basal planes within 10 degrees of perpendicular to the wire axis; the existence of a significant volume fraction of such material would increase the value of diffracted intensity at the 90-degree position to somewhere near the values at the 30 and 150-degree positions. Thus, the determinations of prismatic intensity provide a semi-quantitative measure of the missing information in the basal distribution curve. This information was verified metallographically in the transverse sections; no grains were observed which remained at, or close to, extinction at all stage rotations. (3)

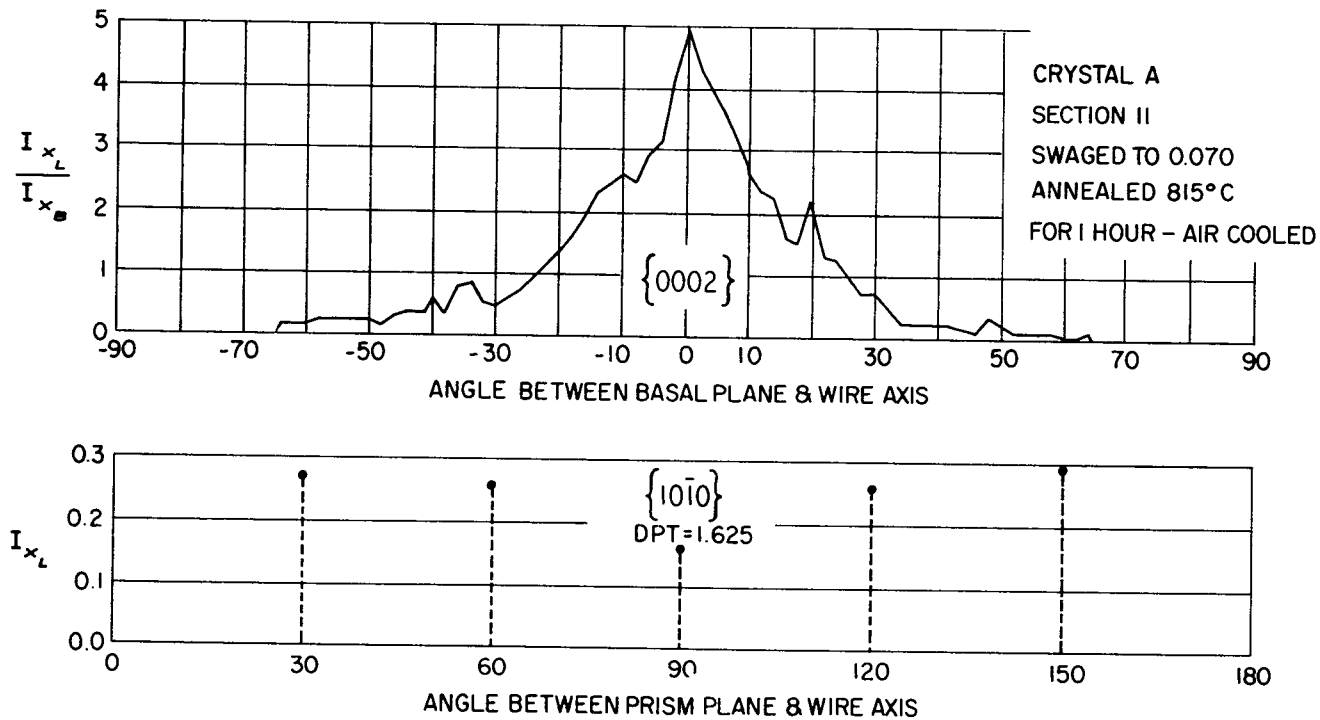


Fig. 7 - Atomic - Population - Distribution - Curve for $\{0002\}$ and Diffracted Intensity at Several Positions for $\{10\bar{1}0\}$ Debye Ring. Sample A. 11 as Swaged and Annealed

The foregoing information indicates that the swaged and annealed product from sample A.11 is virtually identical to commercially-pure beryllium wire (in a comparable condition), with the exception of grain size. The DPT value agrees quite well with comparable values obtained from commercially-pure metal. (8)

Two blanks from crystal 31 were swaged successfully to polycrystalline aggregates. The approximate dimensions of these swagings upon dejacketing and the number of cycles to which they were subjected are:

31 B	5 cycles	0.085 ± 0.010-inch diameter by 27-inches long
31 C	5 cycles	0.085 ± 0.010-inch diameter by 22-inches long

In both cases, the jacketed beryllium was annealed at 815° C for 40 minutes and air cooled after each of the first six swaging passes as well as after each of the first three swaging cycles. An anneal of 730° C for 40 minutes and air cool was used after cycle 4. No anneal was applied after cycle 5.

Specimens from sample 31 C were examined metallographically in both transverse and longitudinal sections. Photomicrographs of these sections are presented in Figure 8. The prominent deformation features are quite comparable to those shown in Figure 4. No evidence of non-metallic inclusions was found in any of the four surfaces (two longitudinal and two transverse) which were examined.

Two blanks from crystal D were subjected to swaging. The swaging practice was different for each blank and from the practices which have been described previously in this Section. This difference from the previous practices was a result of the dimensions of crystal D. In preference to rejacketing so as to continue swaging at a relatively large assembly diameter, it was decided to accomplish without rejacketing an amount of deformation which would be equivalent to three cycles. This was accomplished by slipping the beryllium sample into the first jacket and then passing this assembly through fourteen swaging dies so as to reduce the assembly OD to 0.500-inch diameter without rejacketing. An anneal of 815° C for 1 hour and air cool was applied after each of the first six passes, after the tenth pass, and after the fourteenth pass.

Sample D.1 was swaged at 450° C using the above practice and was then dejacketed for examination. After pickling away the steel with dilute nitric acid, the swaging was measured to be 0.365 ± 0.015-inch diameter

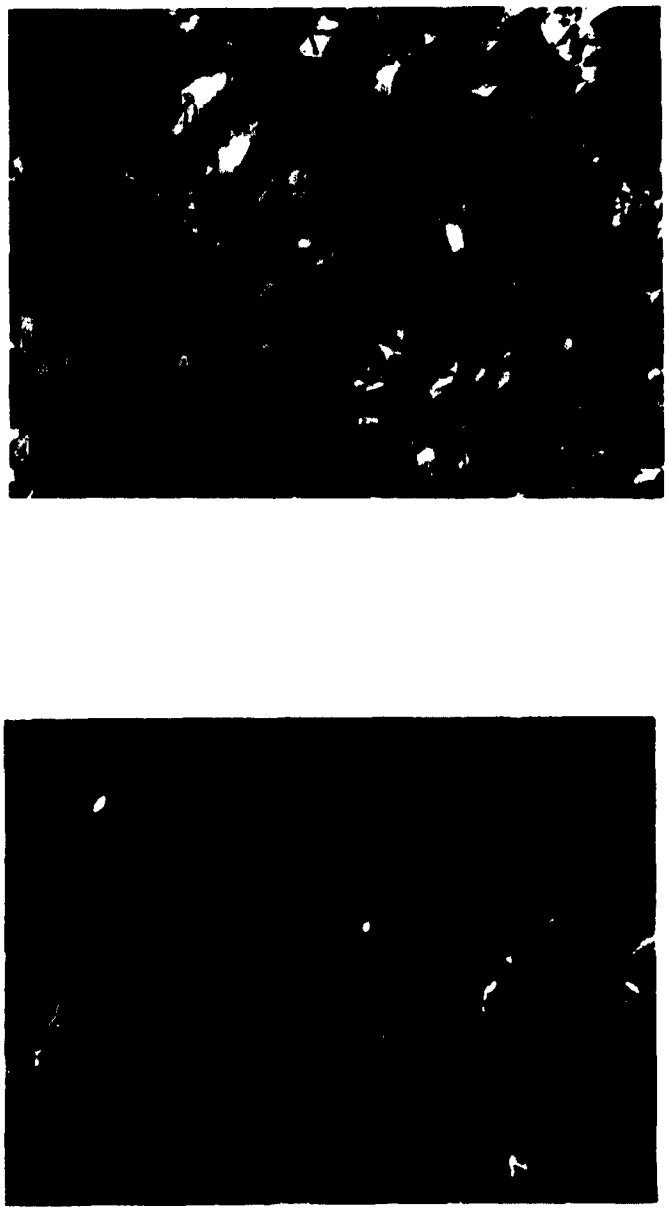


Fig. 8 - Photomicrographs of Sample 31 C
After Swaging

A. Transverse Section; As-Swaged; 100X, PD
B. Longitudinal Section; As-Swaged; 100X, PD

by 9 1/2-inches long. The swaged beryllium appeared to be free of surface defects. This sample was then given a flash pickle in $HF-HNO_3$. This etch revealed numerous cracks which were oriented both transversely and longitudinally. This piece was unsuitable for further processing and was scrapped.

Several transverse sections from sample D. 1 were examined metallographically. The structure was found to be crazed with microcracks. Several very small non-metallic inclusions were found along these cracks, but these inclusions were few and far between.

A Debye camera powder X-ray pattern was obtained from one of these metallographic specimens. The specimen was attritited to powder in a mullite mortar and with a mullite pestle. The powder pattern was indexed in the normal manner. Beryllium lines were identified. A trace amount of mullite was identified. This left but two very weak lines which were tentatively identified as emanating from a trace amount of alpha-quartz. These lines were so weak that it was impossible to index them precisely, and this last identification may be erroneous.

None of the above data was considered to provide an explanation for the break-up of sample D. 1 during swaging.

It was decided to try again with sample D. 2. Upon the recommendation of The Franklin Institute, two changes were introduced in the swaging practice. First, the swaging temperature for the first fourteen passes was increased to 600° C. Second, steel end-plugs were placed at each end of the beryllium crystal so as to restrain the "run-out" of the beryllium. This was the first time that end-plugs were used in this program. The same is true of the 600° C swaging temperature.

Prior to jacketing sample D. 2, both ends of this blank were polished and were examined metallographically. A few, very small non-metallic inclusions were found to be scattered about on each end.

Sample D. 2 was jacketed and swaged to an assembly OD of 0. 500 inch. A second jacket was applied and another swaging cycle was performed. Annealing was the same as in the case of sample D. 1 with the addition of another anneal at 815° C after the last cycle.

The sample was dejacketed as before, and again there were no surface defects observed. The swaged beryllium was approximately 0.285 ± 0.010-inch diameter by 11-inches long at this point. This swaging was then flash-pickled in $HF-HNO_3$. The etching revealed two

transverse cracks which were located about 4 and 5 inches, respectively, from one end. This 5-inch length was discarded, and the remaining 6 inches were cut into three equal pieces which were identified as D. 2a, D. 2b, and D. 2c. Sample D. 2a was at one end of the swaging and sample D. 2c was adjacent to the 5-inch discard, which put this sample almost in the center of the swaging as dejacketed.

Sample D. 2a was rejacketed and reswaged at 450° C without end-plugs. The swaging practice was identical to that used for sample 31 C, except that there were two steel jackets on this sample before the beryllium began to experience deformation during swaging.

Sample D. 2a was subjected to 4 swaging cycles and was dejacketed. The approximate dimensions of this wire were 0.110 $\pm$ 0.010-inch diameter by 15-inches long.

Sample D. 2b was also rejacketed and reswaged at 450° C without end-plugs and in a manner which was identical with that used for sample D. 2a.

Sample D. 2b was subjected to 5 swaging cycles and was dejacketed. The approximate dimensions of this wire were 0.095 $\pm$ 0.004-inch diameter by 22-inches long.

Samples D. 2a and D. 2b were flash pickled in $HF-HNO_3$ and were examined at 30 X for surface defects. None were found.

It should be emphasized at this juncture that just as surely as there is no assignable cause for the failure experienced with D. 1, there is no assignable cause for the success experienced with D. 2. Indeed, if one were to predict from the experience gained in this program prior to the processing of crystal D then the prediction would be that both samples would swage successfully. After the failure of D. 1, changes were instituted without regard to systematic investigation. Rather, any change which might suppress microcrack formation was an acceptable candidate for inclusion in the processing of D. 2. The fact that but two changes were instituted is no more than a reflection of the limited ingenuity of the investigators.

But even this statement is not completely accurate because there were four recognized differences involved: Sample D. 2 was a rather perfect single crystal, not a tricrystal; Sample D. 2 was at a different position with respect to start of zone than was D. 1; the end-plugs were implimented for D. 2; the working temperature was changed.

B. Wire Drawing of Polycrystalline Aggregates

The techniques for this part of the processing were well established prior to the initiation of this program. [1] The plan, here, was to process the swaged, zone-refined metal by existing wire-drawing techniques which had been developed for commercially-pure beryllium.

The wire-drawing efforts on material from crystals A and B were all in vain. This material broke up catastrophically during the wire drawing. Most of the losses were caused by longitudinal splits which opened up upon pickling of the die-entry points and which had to be cropped. Occasionally the wire failed by transverse cracking during the wire-drawing operation. These transverse failures were never the clean, fine-grained tensile fractures as are seen occasionally in commercially-pure beryllium, but were fibrous in appearance such as a broken piece of wood.

Sample A. 4 broke up at 0. 0495-inch diameter.

Sample A. 6 broke up continuously and was destroyed completely upon reaching 0. 02441-inch diameter.

Sample A. 11 broke up continuously and was but 3/4-inch long by the time a diameter of 0. 0257 inch was attained.

Sample B. 3-1 broke up continuously and was destroyed almost completely upon reaching 0. 051-inch diameter.

Sample B. 5-2 was passed through wire dies quite successfully until this wire "rounded-up" at 0. 047-inch diameter. From here on this piece broke up continuously until it was destroyed completely upon reaching 0. 035-inch diameter.

Various combinations of drawing temperature, drawing speed, and annealing practice were tried during the processing of the material from crystals A and B. All to no avail.

Some of the scrap which was generated during the drawing of these samples was utilized to check for chemical contamination from the processing. Pieces from sample A. 6 were used to determine the residual resistance ratio of the drawn wire. One piece was in the unannealed condition and measured 0. 0538-inch diameter by 1-inch long. The other piece was in the annealed condition and measured 0. 0510-inch diameter by 1-inch long. The eddy-current technique was not used in

this instance; a direct contact method was employed. Both of these samples yielded a RRR = 16. This value would be expected to be somewhat lower than the value for the single crystal starting stock (RRR = 17 to 18) due to the introduction of grain boundaries and deformation damage (scattering centers such as vacancies, jogs, and loops). However, the modest decrease from about 17.5 to 16 does not indicate any significant chemical contamination due to the swaging and drawing.

Scrap from sample A.4 was analysed spectrographically. The results of this analysis are presented in Table III along with the results from unprocessed material (A.3 and A.5 which have been reported previously in Table II and are repeated here for facile comparison with processed A.4).

From Table III, it is seen that only in the case of copper is there any indication of contamination. (A thorough review of the swaging and drawing procedures was made but no probable source of copper contamination was found.) It seems reasonable to state that there is little or no chemical contamination caused by the fabrication procedures.

Numerous metallographic examinations were made of transverse and longitudinal sections from drawn wires. Prominent in all of these sections were the stringers of non-metallic inclusions as in Figure 3. These stringers ranged over long distances (more than five wire diameters) in the longitudinal sections.

A forward reflection pin-hole camera pattern was produced from the 3/4-inch length of sample A.11 in the as-drawn condition at 0.0257-inch diameter. This wire had been reduced 69% in cross-sectional area from the last anneal. A negative reproduction of this pattern is presented in Figure 9. Note the striations in the Debye rings. The relative intensity of the three beryllium oxide Debye rings (three closely spaced rings just inside the $\{10\bar{1}0\}$ ring for beryllium) may be out of proportion to the beryllium Debye rings, because a long exposure time was used intentionally to bring out indications of minor phases. These oxide rings show no epitaxy with the beryllium but they are striated. Since the surface of this specimen had been cleaned thoroughly and flash pickled with $HF\text{-}HNO_3$, it is felt that this beryllium oxide was located internally in the specimen. It seems probable that these BeO indications emanate from the non-metallic stringers which were seen in the photomicrographs. It was believed that these BeO stringers were the principal reason for the lack of drawability of the material from crystals A and B. Drawing effort on crystals A, B, and C was terminated for this reason.

TABLE III

CHEMICAL ANALYSIS OF SCRAP GENERATED DURING THE WIRE DRAWING OF SAMPLE A.4[a]

Element	Analysis (ppm) and date of analysis of: A.3[b] 4/27/62	A.4[b] 6/8/62	A.5[b] 4/27/62
C	--	--	--
Al	12	-30	10
Cr	8	8	8
Fe	70	50	85
Mg	-25	-30	-25
Mn	-6	-6	-6
Ni	90	90	60
Ti	-6	-6	-6
Cl	--	--	--
Ca	-85	-85	-85
Co	-3	-3	-3
Cu	20	120	25
Si	70	55	55
Zn	-55	-55	-55

[a] Sample converted to BeO; results are on a Be metal basis. Emission Spectrography.

[b] Minus sign indicates "less than".

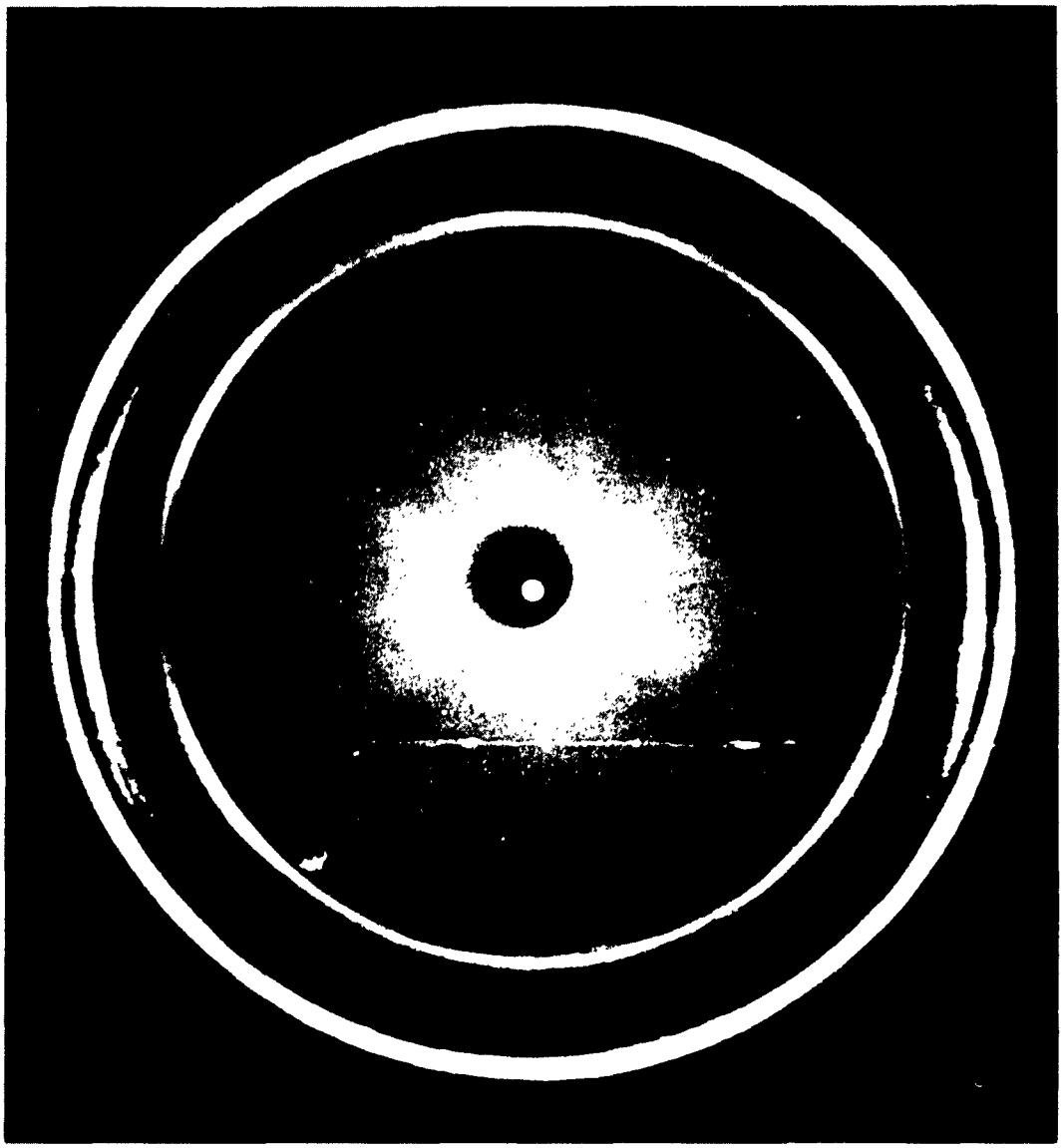


Fig. 9 - Forward Reflection Pin-Hole Camera Pattern for
A.11 Drawn to 0.0257-Inch Diameter.
(Wire Axis is Vertical)

The pattern in Figure 9 was analysed for photodensity as before and the reduced data are shown graphically in Figure 10. The basal plane distribution looks much as a comparable distribution for commercially-pure beryllium; the major difference being the jagged peaks which are indicative of the presence of at least three very large grains. The value of the DPT parameter is exactly equal to comparable values of this parameter from commercially-pure metal.(8) The five values of reflected intensity similarly match those for commercially-pure metal in their ratios to each other.

This piece of wire was annealed at 815°C for 70 minutes and was air cooled. The annealed wire was cleaned as before. A diffraction pattern was produced for the annealed wire. The reduced data from the photodensity measurements are presented in Figure 11.

Here, for the first time in this work, the high-purity beryllium has exhibited a behavior which is markedly different from commercially-pure materal. The DPT value after heat treatment and the value of the degree of destruction of $\langle 10\bar{1}0 \rangle$ fibering* are identical with comparable values(8) from commercially-pure metal. But the amount of reorientation of basal planes with respect to the wire axis is unanticipated from results obtained using commercially-pure beryllium. Indeed, the basal distribution in Figure 11 is indistinguishable from patterns emanating from block-pressed, commercially-pure material which has not been wrought. This is not a shift in the direction of the response to heat treatment but is a much larger degree of response than has been observed for commercially-pure material. This dramatic response suggests that the grain boundaries have a very high mobility in the zone-refined beryllium, at least in the volume from which Figure 11 was generated.

The transverse grain structure of this wire in both the as-drawn and annealed conditions is best described as a duplex structure. There appeared to be grains of essentially two distinct sizes with virtually no grains in the intermediate size range. The two average grain diameters were estimated by the comparison technique. An estimate was made of the area fraction of the optical surface which was populated by grains of each size. An average grain diameter was calculated.

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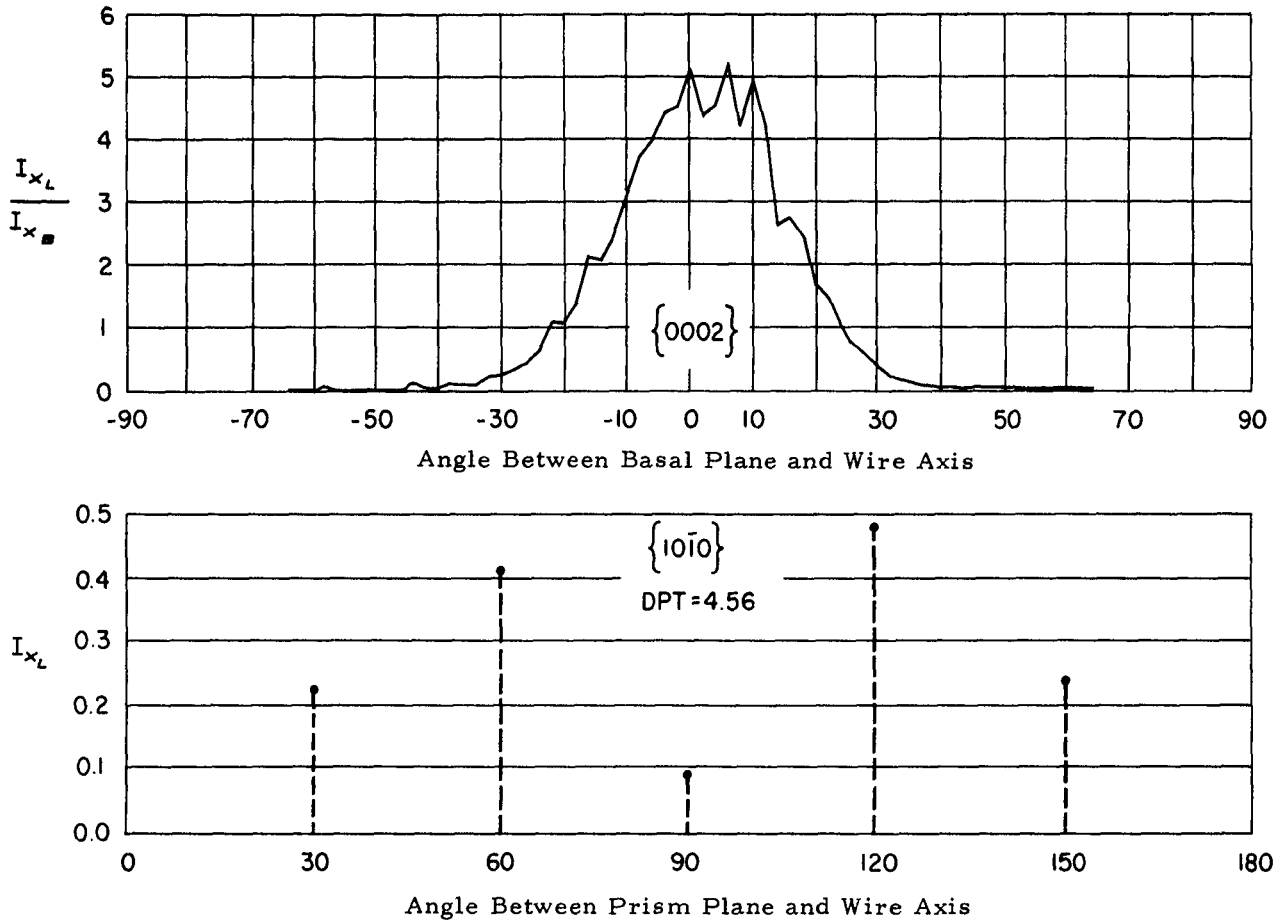
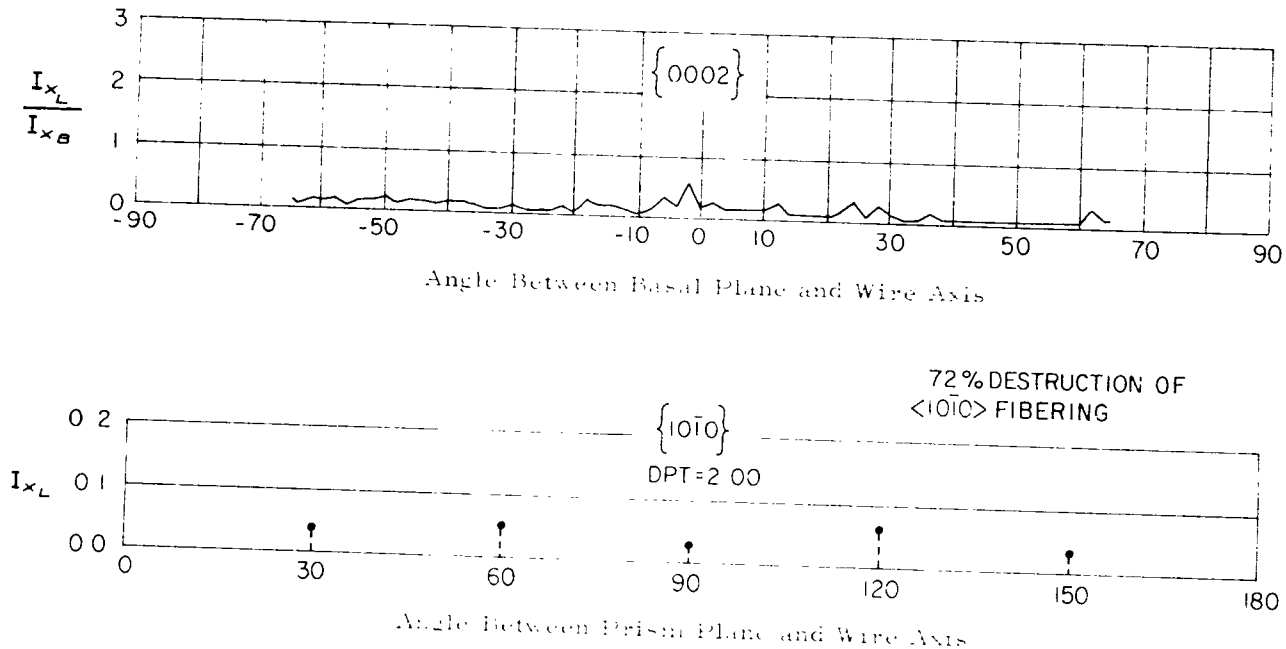
*The value of the DPT before heat treatment was 4.56. If this value had been reduced to 1.00 by heat treatment, then there would have been 100% destruction of $\langle 10\bar{1}0 \rangle$ fibering; actually the value was reduced to 2.00 by heat treatment which corresponds to $(2.56 \div 3.56)\ 100\% = 72\%$ destruction.



Crystal A Section 11

Swaged to 0.070 and Annealed 815°C - 1 Hour-Air Cool
Drawn to 0.060 " " " " " "
" to 0.054 " " " " " "
" to 0.046 " " " " " "
" to 0.0392 Not Annealed
Redrawn to 0.0335 " "
" " 0.03176 " "
" " 0.02859 " "
" " 0.02712 " "
" " 0.02573 " "

Fig. 10 - Atomic - Population - Distribution - Curve for {0002} Debye Ring and Diffracted Intensity at Several Positions for {10$\bar{1}$0} Debye Ring. Sample A.11 as Drawn to 0.0257-Inch Diameter



Same as Figure 10 Plus Annealed at 815°C for 70 Minutes and Air Cooled

Fig. 11 - Atomic - Population - Distribution - Curve for {0002} Debye Ring and Diffraction Intensity at Several Positions for {10$\bar{1}$0} Debye Ring. Sample A.11 as Drawn to 0.0257-Inch Diameter and Annealed.

The as-drawn wire was estimated to have a 50% area fraction populated by 72-micron grains and a 50% area fraction populated by 18-microns. After annealing, the wire was estimated to have a 50% area fraction populated by 67-micron grains and a 50% area fraction populated by 26-micron grains. The overall average grain size in each instance is:

0.0257-inch diameter (A.11); as-drawn; 45-micron

0.0257-inch diameter (A.11); annealed; 47-micron

It is interesting to note that although the overall grain size was virtually unaffected by heat treatment, a substantial structural change did occur.

This annealed piece of 0.0257-inch-diameter wire from sample A.11 was tensile tested at room temperature. No reduced section was produced for a gage length; the 3/4-inch-long wire was gripped in drill chucks with a 0.250-inch separation between the chucks which defined the test gage length. An initial strain rate of 0.10 in./in./min was used. The following reduced data were obtained from this test:

Engineering Yield Strength (0.2% offset)	23.43 ksi
Engineering Tensile Strength	37.95 ksi
Elongation (from load-deflection chart)	6.05%
Tensile Reduction in Area (at the fracture)	6.83%
Young's Modulus (from load-deflection curve)	44.2×10^6 psi
Engineering Ultimate Strength	30.43 ksi

Fracture occurred near the center of the gage length.

Due to the random distribution of basal planes in this specimen, these results are best compared not against commercially-pure beryllium wire but against commercially-pure, block-pressed beryllium. This comparison shows that the yield and tensile strength values from the above test are about 60% of values for block-pressed material but that the ductility is 3 to 4 times higher. Perhaps more important is the fracture characteristic.

The load decrease following the maximum load was not a necking result. This is attested by the fact that the ultimate strength was lower than the tensile strength. The load-deflection curve between maximum load and breaking load was noticeably jagged. This suggests that the grains fractured sequentially with an appreciable amount of deformation

subsequent to individual failures. Such a discontinuous failure would be consistent with the presence of incoherent non-metallic inclusions shown in Figure 3. These inclusions would weaken inherently the wire but would also impede crack propogation between certain grains.

The preferred orientation of the annealed wire (A.11) was re-evaluated after tensile testing. The forward-reflection-pin-hole-camera pattern showed a modest amount of asterism, as would be expected from a strained specimen. The pattern was produced from the volume of the wire immediately adjacent to the tensile-fracture surface. The basal plane orientation distribution is presented in Figure 12. Note that both halves (separated by the wire axis direction on the pattern) of the basal Debye ring have been analysed in this instance. The distributions in Figure 12 compare to Figure 11. The distributions in Figure 12 are not as random as that in Figure 11 but are significantly more random than that in Figure 10. The higher intensities in Figure 12 as compared to Figure 11 may be the result of increased scattering power due to the straining of the lattice. Alternatively, the volume fraction of the wire from which Figure 12 was obtained was not the same as that from which Figure 11 was obtained. It might well be that certain portions of the wire were more highly randomized than others by the anneal. In any event, the reorientation caused by annealing was unexpectedly high.

The swaged polycrystalline aggregates from crystal 31 were subjected to wire drawing at 450° C. The general procedure was identical to that used for crystals A and B. A 10% reduction-in-area-per-pass schedule was used throughout. Only one "process anneal" was employed; this anneal was applied as soon as the variations in diameter (which are inherent to the swaging practice) were "ironed-out" by wire drawing. The anneal was applied when the wire was at a diameter of 0.076 inch; the anneal consisted of heating to 815° C, holding for 30 minutes, and air cooling. The drawing was then resumed with no subsequent "process annealing".

Sample 31 B was drawn without fracture * to a finished diameter of 0.0105 inch. Specimens for tensile testing were cropped at 0.01367-inch diameter, 0.01297-inch diameter, and at finished size. No limit of drawability was found, and the choice of finished diameter was completely arbitrary.

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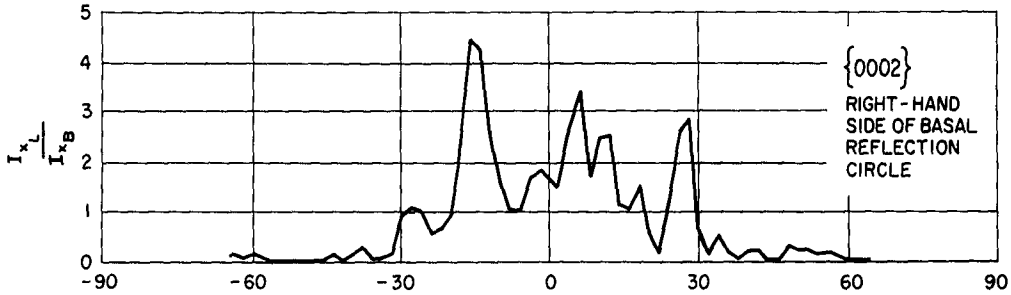
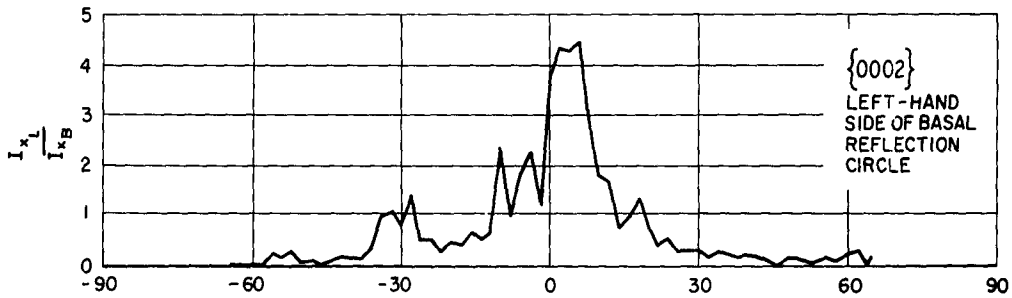
*"Without fracture" does not include several spurious breaks which were unambiguously assignable to processing defects such as bad dies or poor lubrication.



Angle Between the Basal Plane and Wire Axis

Note: 1. X-Ray Beam was Incident on the Sample at a Location Which was Immediately Adjacent to the Tensile Fracture Surface.

2. Same Condition as in Figure 12, Progress Report No. 1, Plus Room Temperature Tensile Testing to Fracture



Angle Between the Basal Plane and Wire Axis

Fig. 12 - Atomic - Population - Distribution - Curve for {0002} Debye Ring. Sample A.11 after Tensile Testing to Fracture at Room Temperature

Sample 31 C was drawn without fracture* to a diameter of 0.00727 inch. The processing schedule called for a fresh coat of lubricant prior to passing the 0.00689-inch-diameter die. The draw force is known to be abnormally high during the first pass on a fresh coat of lubricant and, in the case at hand, it was high enough to exceed the tensile strength of the drawn wire. Several fractures were encountered before a 24-foot length was passed successfully. This length was then redrawn without fracture* to 0.00407-inch diameter without additional application of lubricant. Specimens for tensile testing were cropped from sample 31 C material at diameters of 0.02573 inch, 0.01297 inch, 0.00727 inch, 0.00477 inch, and at finished size. No limit of drawability (except for the lubrication limitation) was found, and the choice of finished diameter was dictated by the smallest die on hand at the time.

The 0.02573-inch-diameter test material which was produced from sample 31 C was cut into four tensile specimens and four metallographic specimens. Forward-reflection, pin-hole-camera patterns were obtained in the as-drawn condition from the center of the gage length of each tensile specimen. Two of the tensile specimens were then heat treated to compare with the single tensile specimen at this diameter from crystal A.11 (815°C for 60 minutes and air cool). X-ray diffraction patterns were obtained after heat treatment from the center of the gage length of each of these two annealed specimens.

These patterns, none of which showed any discernible indications of BeO, were analysed for basal plane orientation (angle between the $\{0002\}$ and the wire axis) distribution. The orientation distributions for the "as-drawn" condition are presented in Figure 13.

The orientation distribution after heat treatment of one tensile specimen is presented in Figure 14. As will be shown, the heat-treated structures were extremely coarse grained. The X-ray pins were not rotated about the wire axis (nor about any other axis) during exposure, and thus the chances of having very many, if any, grains positioned (in a rotational sense about the wire axis) for basal plane diffraction were slim. Indeed, one of the two heat-treated tensile specimens produced a pattern which contained no discernible evidence of basal plane diffraction!

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*Refer to footnote on page 32

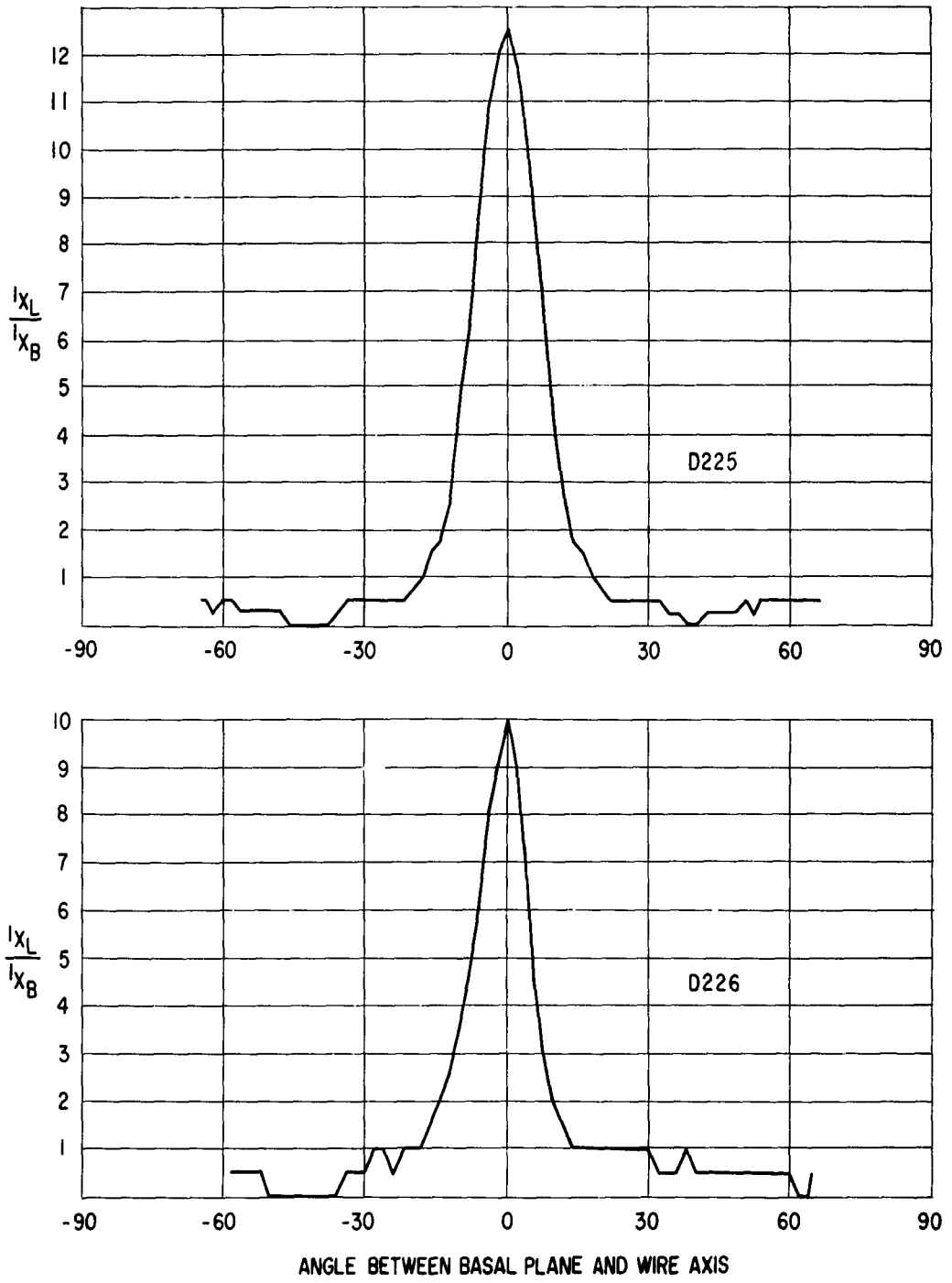


Fig. 13 - Atomic - Population - Distribution - Curves for {0002} Debye Ring: As-Drawn Wire from Sample 31 C at 0.02573-Inch Diameter; Four Determinations

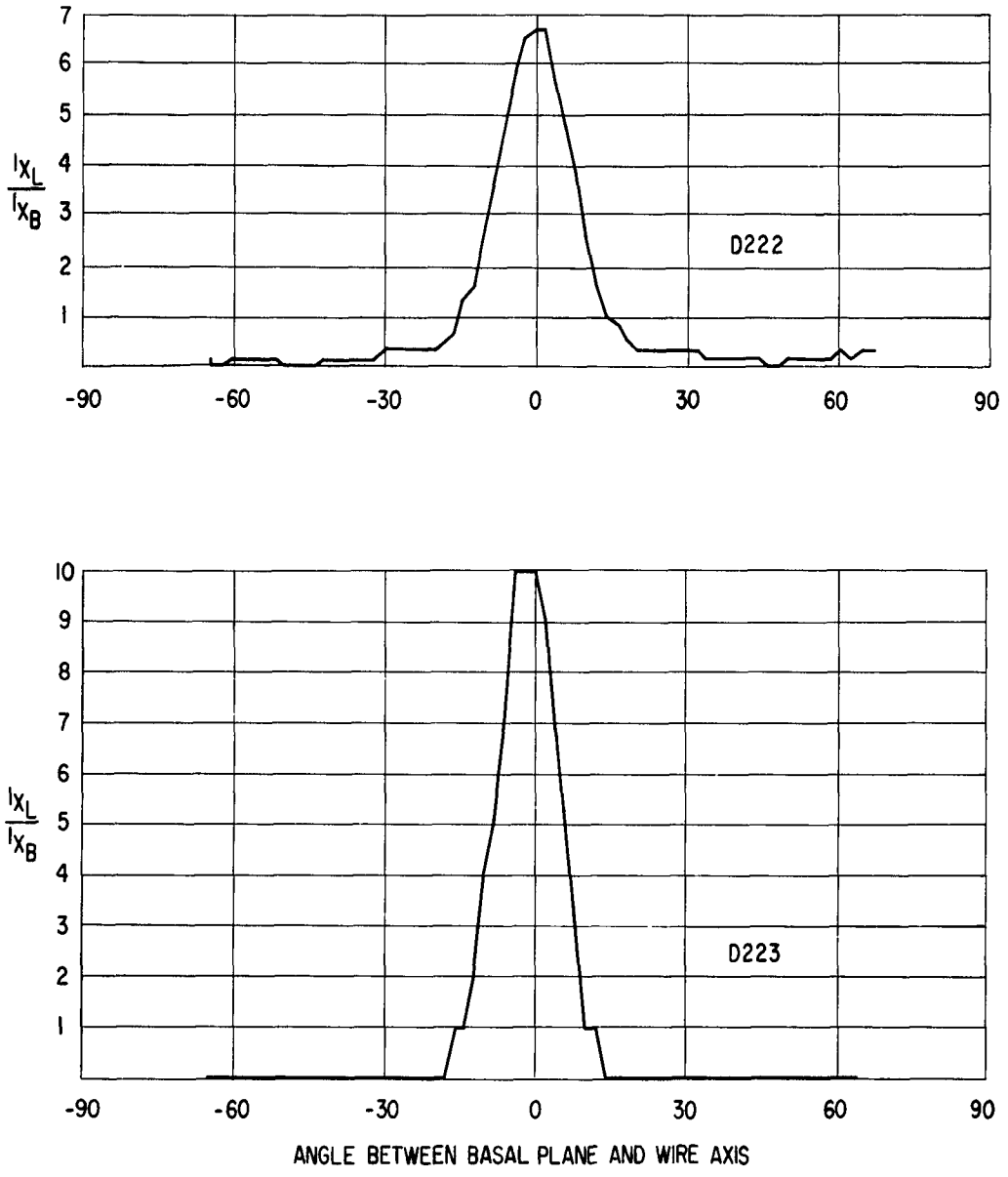


Fig. 13 (Cont.) - Atomic - Population - Distribution Curves for {0002} Debye Ring: As-Drawn Wire from Sample 31 C at 0.02573-Inch Diameter; Four Determinations

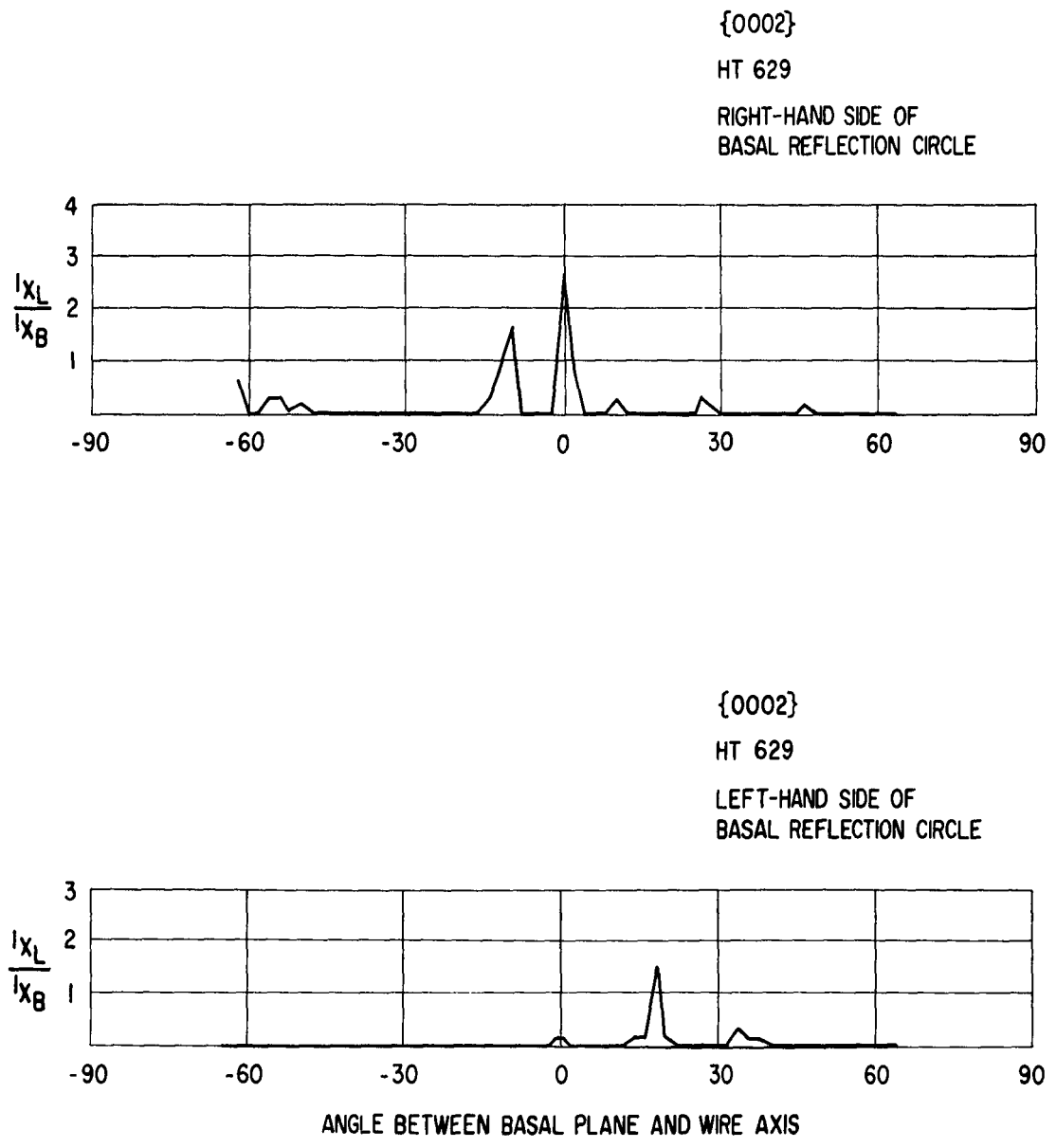


Fig. 14 - Atomic - Population - Distribution - Curves for {0002} Debye Ring: Wire at 0.02573-Inch Diameter from Sample 31 C; Heat-Treated Condition

The basal orientation distributions of the heat-treated specimens after tensile testing (room temperature) are presented in Figure 15. These patterns were produced from a volume of material which was immediately adjacent to the tensile fracture surface. No indications of BeO were found.

The orientation distributions for the as-drawn wire were indistinguishable from distributions for as-drawn commercially-pure wire (of equivalent processing history). Interpretation of the heat-treated distributions is a bit difficult due to the large grain size. Figure 14 shows some indications of a tendency to randomize much as did the wire from crystal A (at this diameter), but it is dangerous to draw a firm conclusion without having a considerably larger number of grains contributing to the distribution. It would really be best to repeat this heat treating at one or more temperatures which are well below 815° C and/or for shorter times at temperature so as to avoid extreme grain growth.

The "as-drawn" microstructure of the 0.02573-inch-diameter wire from sample 31 C is presented in Figure 16. The average transverse grain size was estimated to be 14 microns for the transverse section in Figure 16, and to be 12 microns for the other transverse section which is not shown in Figure 16. The sections presented in Figure 16 are distinguishable from comparable sections of commercial metal, only in that the wire from zone-refined metal showed none of the spherical oxide particles which are characteristic to commercial metal; neither was there any evidence of the non-metallic stringers which were observed in wire from crystals A and B.

The "heat-treated" microstructure of the 0.02573-inch-diameter wire from sample 31 C is presented in Figure 17. The inordinately large grain in Figure 17 B did not contain discernible low-angle boundaries.

The amount of grain growth which was encountered here was completely unanticipated. Heavily worked (natural strain induced by wire drawing equal to 2.5) commercial wire has been found to change from 6-micron transverse grain size to 13-micron transverse grain size during heat treatment at 850° C for 100 minutes. (8) The wire from crystal A (natural strain induced by wire drawing of 1.16) which was heat treated the same as the wire from sample 31 C showed the small grains to change from 18 microns to 26 microns, while the large grains of this duplexed structure suffered very little change in size. The high degree of grain-boundary mobility in the sample 31 C material is probably indicative of solid solution impurity concentrations which are appreciably lower (9, 10, 11, 12) than have been previously used in this

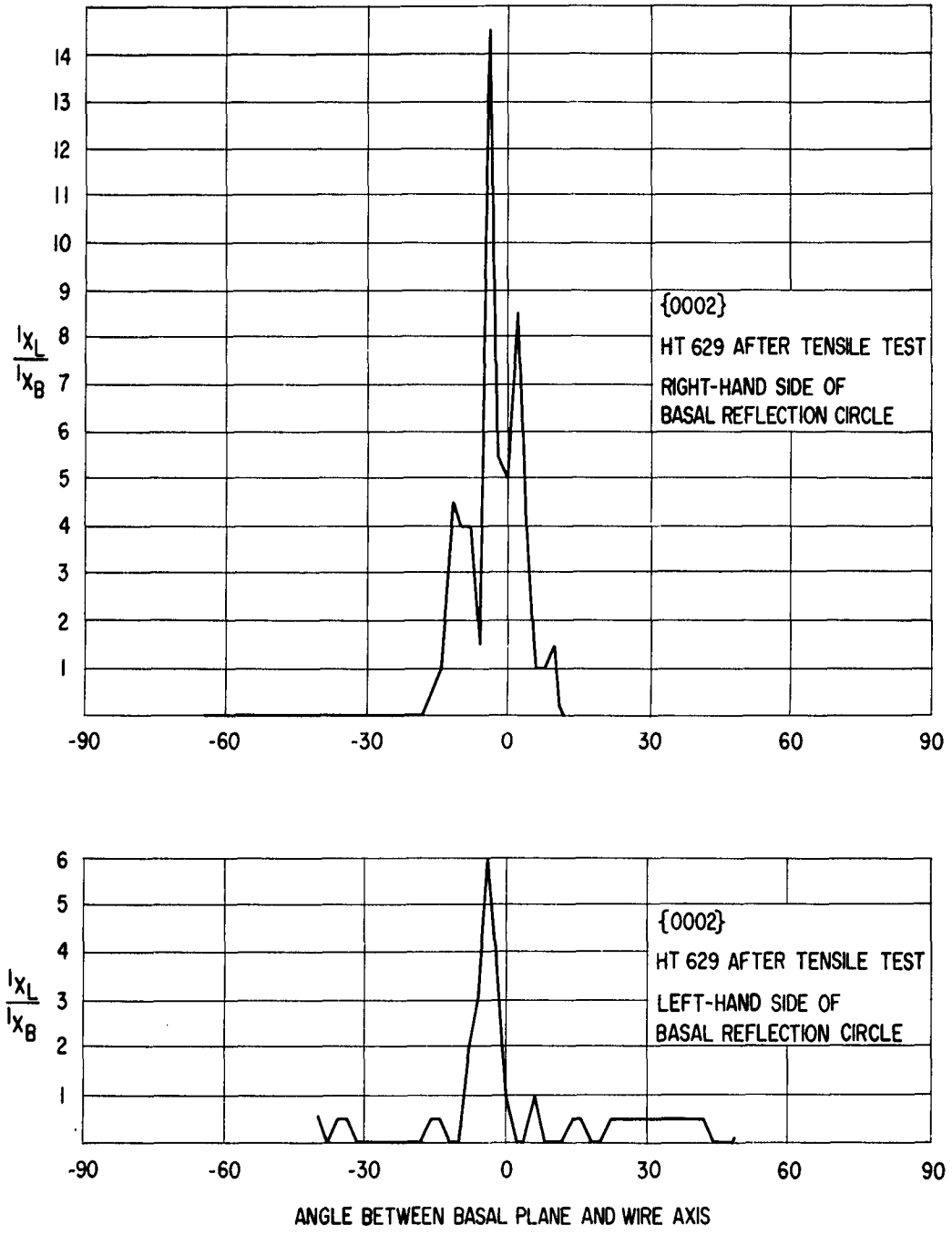
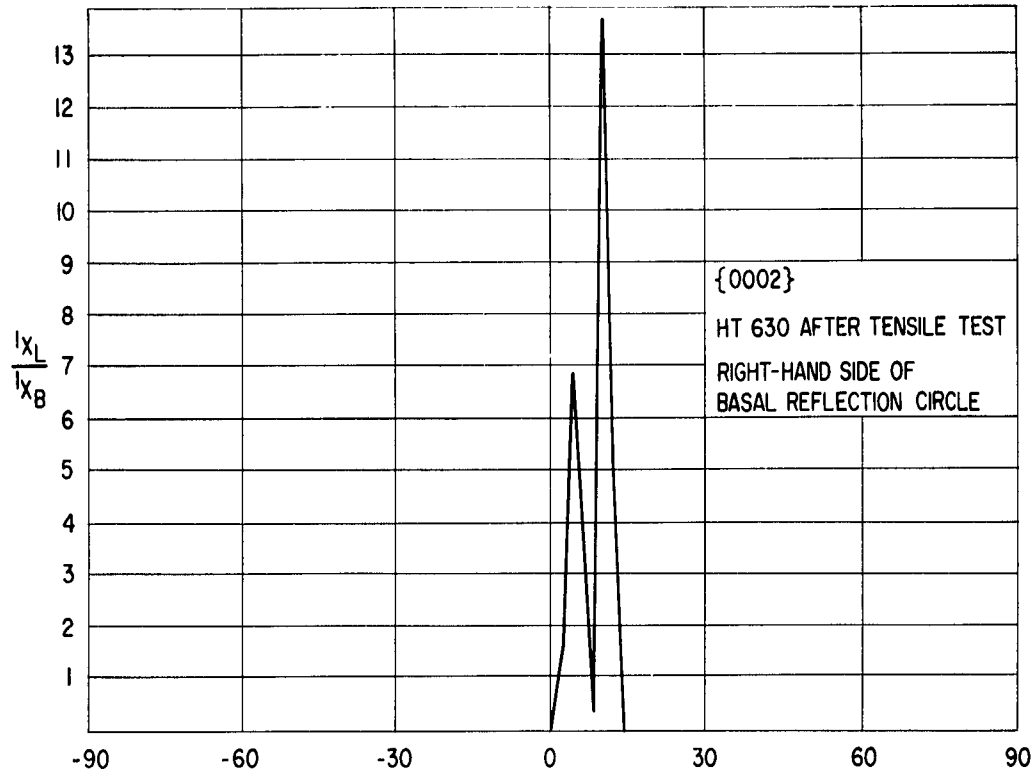


Fig. 15 - Atomic - Population - Distribution - Curves for {0002} Debye Ring: Wire at 0.02573-Inch Diameter from Sample 31 C; Heat Treated and Tensile Tested



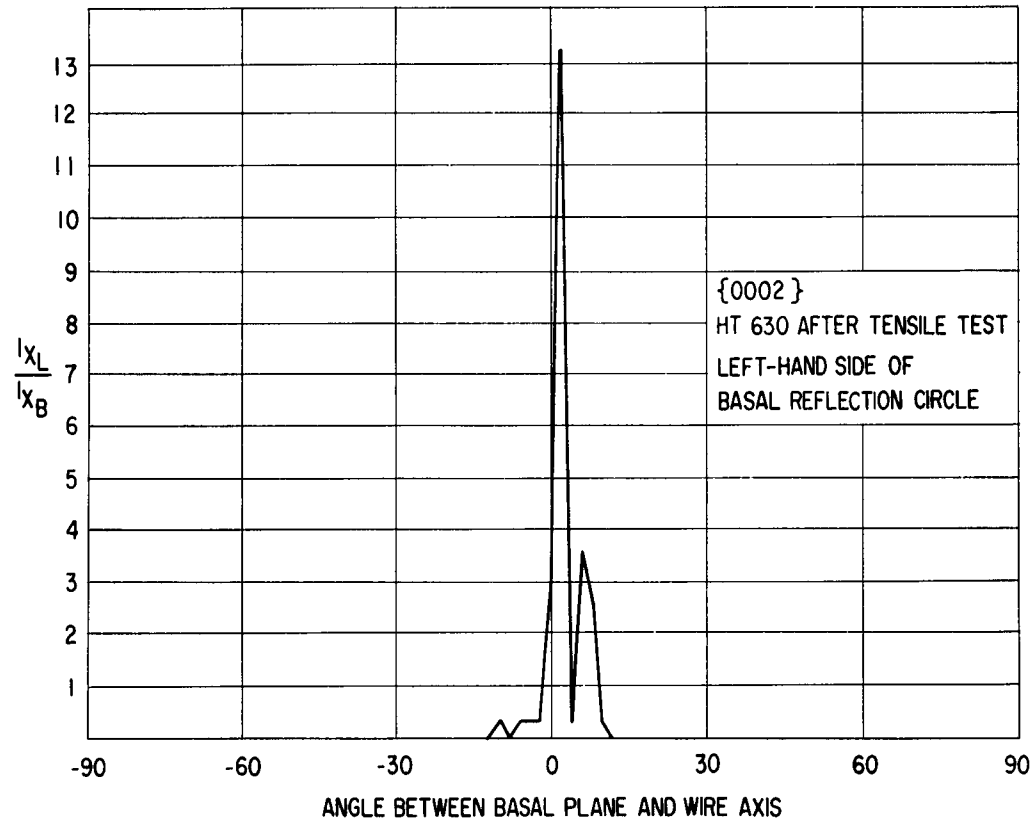


Fig. 15 (Cont.) Atomic - Population - Distribution Curves for {0002} Debye Ring: Wire at 0.02573-Inch Diameter from Sample 31 C; Heat Treated and Tensile Tested

A

B

Fig. 16 - Photomicrographs of Wire at 0.02573-Inch Diameter from Sample 31 C; As-Drawn Condition.

A. Transverse Section; 100X, PD

B. Longitudinal Section; 100X, PD

A

B

Fig. 17 - Photomicrographs of Wire at 0.02573-Inch Diameter from Sample 31C; Heat-Treated Condition

A. Transverse Section; Same as Fig. 16A Plus Heat Treatment (815°C for 60-Minutes and Air Cool); 100X, PD

B. Same as A After Grinding Off About 0.01 Inch from the Surface Shown in A; 100X, PD

program. This is not to say that the total impurity concentration is particularly low, but only that these impurities are well aggregated in one or more ways which do not impede boundary migration as effectively as do supersaturated solid solutions, clusters, and/or early-stage GP-zones. Of course, there is still the possibility that wire from crystals A and B would have exhibited high mobility grain boundaries if the non-metallic stringers had not been present. This possibility has the consequence that these stringers be quite effective in pinning the grain boundaries. It is possible that sample A.11 underwent considerable grain growth during recrystallization and that the final grain size was a measure of the average distance between stringer-barriers.

The authors were unable to locate any data on the room-temperature mechanical properties of very coarse grained powder - metallurgically produced beryllium (with a comparable thermo-mechanical history). Thus, the tensile results obtained from the annealed 0.02573-inch diameter wire from sample 31 C will be compared to those of wrought, coarse-grained commercially-pure cast metal.

Figure 18e, page 491 of reference (5) presents the annealed (800°C for 60-minutes) structure of an extrusion which was made from cast metal. This structure is quite comparable to that which is shown in Figure 17A of this report. Room-temperature tensile properties for extruded cast metal which was annealed at 800°C for 60 minutes have been reported to be: (13)

Test Direction	Tensile Strength ($\times 10^3$ psi)	Elongation (%)
Longitudinal	40.0	1.8
Transverse	16.5	0.18

The longitudinal value may be compared to the tensile results for the 0.02573-inch-diameter (annealed) wire from sample 31 C which are presented in Table IV. The strength levels appear to be about the same; the ductilities may or may not be significantly different.

The propriety of this comparison is still quite questionable. Even though the grain sizes are about the same, one might anticipate that, for instance, a grain size of 100 microns in 0.025-inch-diameter wire is a far more serious affair than the same grain size in a 0.250-inch diameter tensile bar. In other words, the ratio of grain diameter to specimen diameter may be a more meaningful parameter with respect to ductility than is the grain diameter alone. In addition, there is some doubt as to

TABLE IV

ROOM-TEMPERATURE TENSILE RESULTS FOR ANNEALED WIRE FROM SAMPLE 31 C[a]

Drawn Diameter (in.)	Annealing Time[b] (min)	No. of Tests	Average Yield Strength[c] ($\times 10^3$ psi)	Average Tensile Strength ($\times 10^3$ psi)	Average Elongation in 1-inch (%)
0.02573	60	2	25.8	44.5	3.55
0.00727	30	7	25.9	37.3	5.05
0.00654	30	12	20.9	27.2	3.84

[a] 1.00-inch gage length
0.10-inch/inch/minute strain rate
Specimens were lightly etched in dilute $HF-HNO_3$ prior to testing.

[b] Specimens were held at 815°C for the indicated time and air cooled.

[c] At 0.2% offset.

whether cross sections which have a grain structure such as in Figure 17 B will deform cooperatively as a polycrystalline aggregate or non-cooperatively as a complexly stressed single crystal.

Comparison of this annealed 0.02573-inch-diameter wire with same size annealed wire from crystal A shows the two to be reasonably equivalent. The wire from sample 31 C may be slightly stronger and a bit less ductile; no firm conclusion is drawn due to the absence of information as to the precision of these tests.

Table IV presents information for two other drawn diameters which have been annealed and tested. These results are included for information purposes only and are not considered to be interpretable, since no orientation and metallographic characteristics are available for them.

Tensile testing in the "as-drawn" condition was performed on a variety of diameters of wire from samples 31 B and 31 C. The results of this testing are presented in Table V. The information on yield strength and on tensile strength is organized graphically on logarithmic coordinates in Figures 18 and 19. Also included in Figures 18 and 19 are the yield and tensile characteristics of a commercially-pure lot of beryllium*.

Random sampling of the zone-refined metal at each of the sampled wire-drawn strains was highly impractical, since it was of primary interest to ascertain the drawability of this metal and of secondary interest to measure precisely its tensile characteristics. Random sampling would have required numerous subdivisions at each sampled strain. The redrawing of the resulting pieces to produce subsequent sampled strains would not have yielded drawability results of very much significance in that an appreciable volume fraction of the material would have been lost in points and test samples.

Consequently, the samples which were tensile tested differed not only in wire-drawn strain, but quite probably in composition. The composition gradient may be either discontinuous or continuous with the values of sampled strain. This confounds, to some extent, precise interpretation of the plots in Figures 18 and 19. Such a confounding possibility is indicated in these figures by the inclusions of both a shaded band and a solid curve.

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* These data were taken from unpublished work which was performed at and under the sponsorship of The Brush Beryllium Company.

TABLE V

ROOM-TEMPERATURE TENSILE RESULTS FOR AS-DRAWN WIRE FROM SAMPLES 31 B AND 31 C[a]

Crystal	Wire Drawn Strain[b]	Wire Diameter (in.)	Average Yield Strength[c] ($\times 10^3$ psi)	Average Tensile Strength ($\times 10^3$ psi)	Average Elongation in 1-inch (%)	No. of Tests
31 C	2.175	0.02573	48.90	55.25	0.6	2
31 B	3.435	0.01367	81.77	95.27	0.4	6
31 C	3.54	0.01297	93.5	117.6	1.5	6
31 B	3.54	0.01297	82.0	92.3	0.4	11
31 B	3.96	0.01051	94.7	116.2	1.0	5
31 C	4.65	0.00727	105.8	138.7	6.5	29
31 C	5.59	0.00477	106.1	131.7	3.7	28
31 C	5.85	0.00407	107.6	132.5	2.3	10

[a] 1.00-inch gage length
0.10 inch/inch/minute strain rate
Specimens were tested with the wire-drawing lubricant unremoved.

[b] Measured from last anneal; $\bar{\varepsilon}$ = Ln (reduction ratio).

[c] At 0.2% offset.

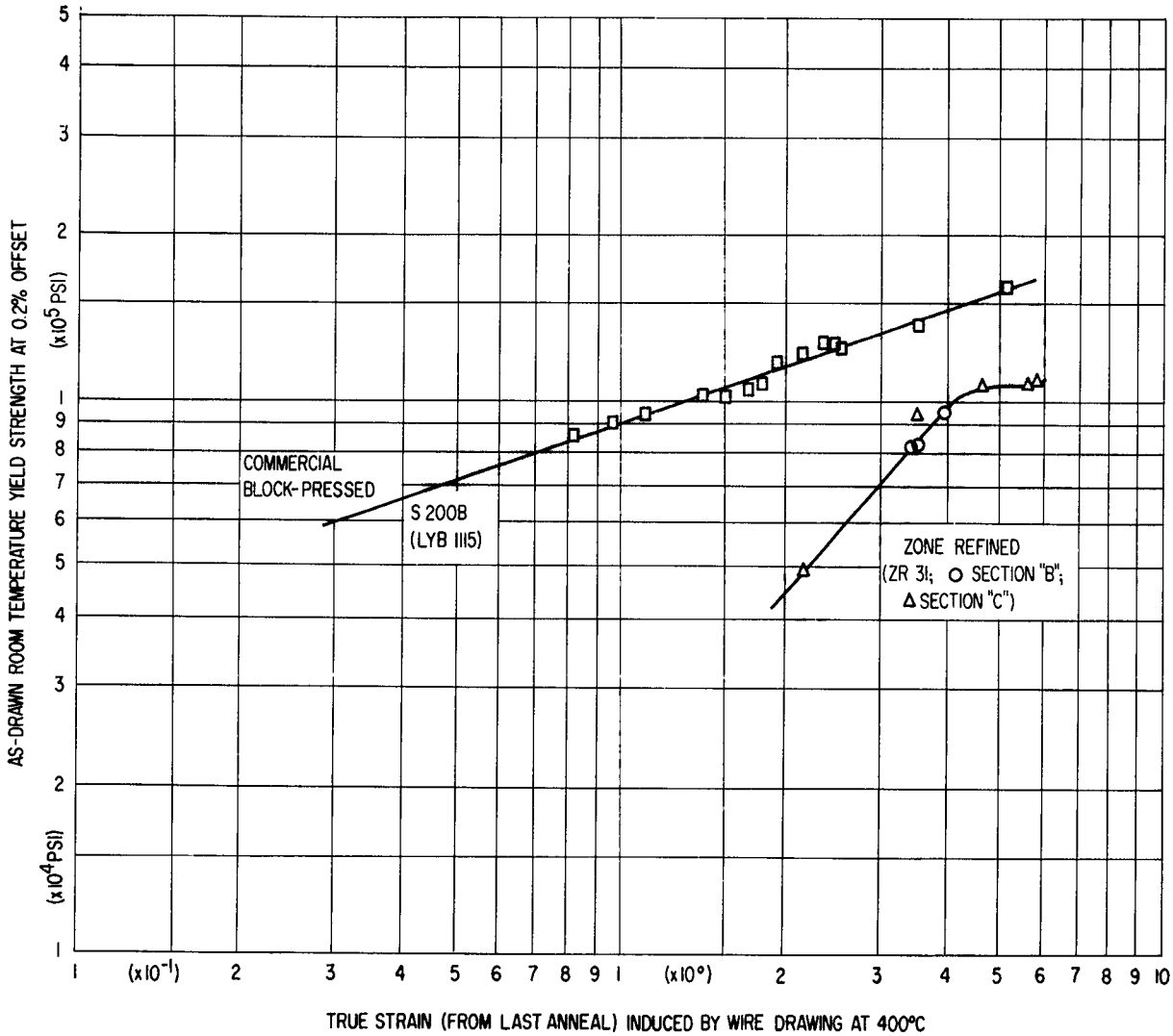

Fig. 18 - The Variation in As-Drawn Yield Strength with Wire-Drawing Strain

Notes: 1. Solid Lines are Drawn Assuming Little or No Sampling Error

2. Bands are Drawn Assuming Large Sampling Error

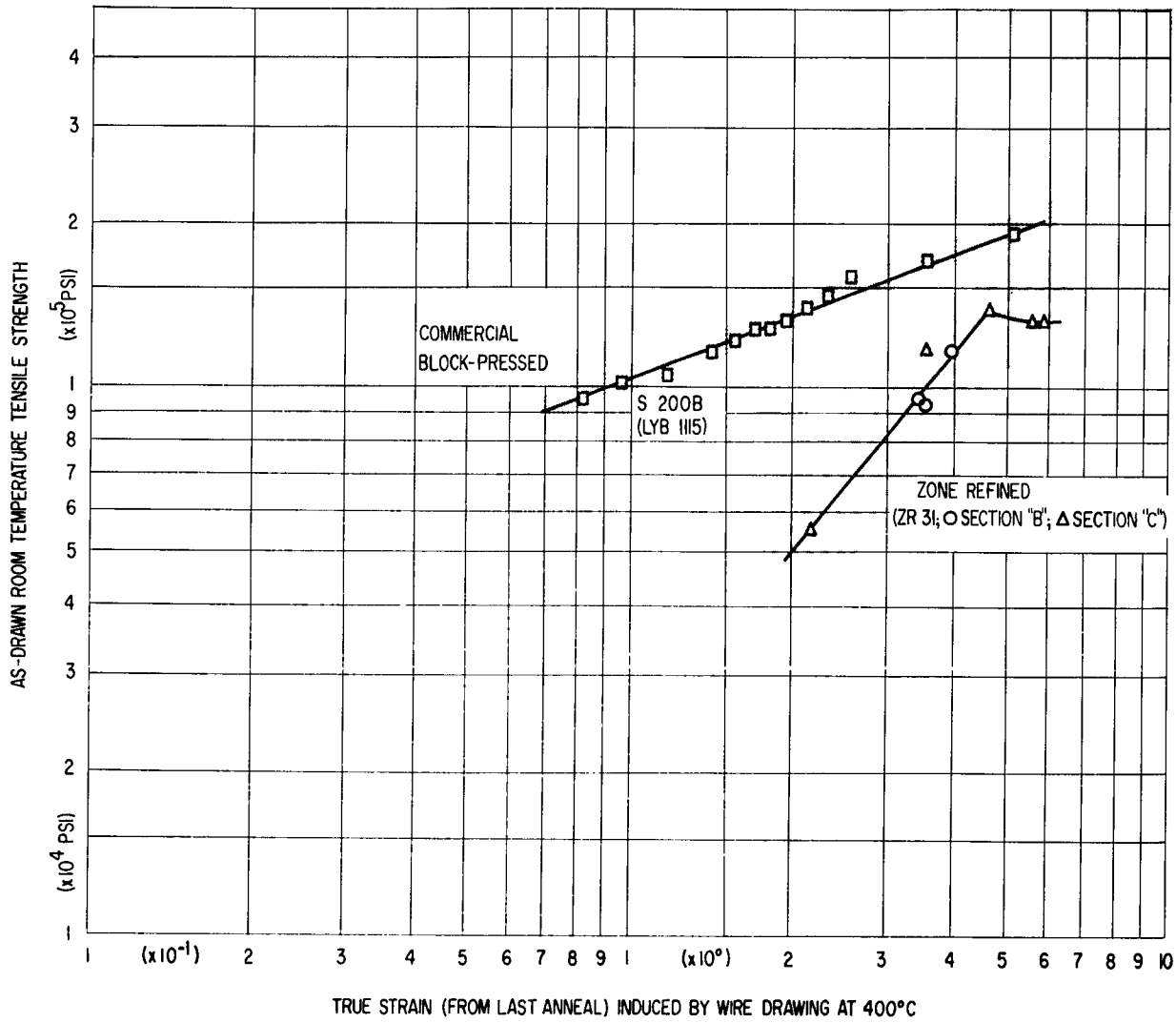

Fig. 19 - The Variation in As-Drawn Tensile Strength with Wire-Drawing Strain

Notes: 1. Solid Lines are Drawn Assuming Little or No Sampling Error

2. Bands are Drawn Assuming Large Sampling Error

Note also that if one discounts heavily the zone-refined data for the lowest sampled strain (strain = 2.175; wire diameter = 0.02573-inch), then the solid curves could be drawn straight and approximately parallel to the solid curves for commercial metal. The confidence in such straight lines would be somewhat lower than that in the commercial curves.

The inflections which are drawn in the solid curves for zone-refined metal could be a sampling accident. If so, this zone-refined metal possesses a large amount of variability as is suggested by the shaded band. More probable is the interpretation of strain-induced recovery at the working temperature.

However, it is possible to draw with high confidence what is probably the most important conclusion with respect to strength. That is, zone-refined beryllium, which is quite soft (or weak as the reader prefers) as a single crystal, can be strengthened through metalworking to become structurally useful.

This conclusion is not as dramatic as it might seem at first. The zone-refined crystals are softer than commercially-pure, cast-metal crystals, primarily with respect to basal glide. Evidence published so far indicates very little, if any, alteration by zone-refining in the prismatic-glide character at room temperature. [14, 15] Metalworking, and certainly wire drawing, develops a polycrystalline aggregate with a preferred crystallographic orientation which, in beryllium, suppresses basal-glide activity and promotes prismatic-glide activity in the majority of structural applications. Thus, the characteristic governing the onset of plastic behavior at room temperature in beryllium mill forms is the characteristic which is unaltered essentially by zone-refining. This rationalization is restricted, primarily, to the yield strength; the tensile strength and the ductility may or may not be more sensitive to other parameters such as the grain size and the resolved normal stress for prismatic cleavage.

Returning to Table V, the room-temperature ductility of the as-drawn wire follows the same general trend with wire-drawn strain as do the strength levels. Commercially-pure, block-pressed beryllium does not show a significant trend but remains, on the average, fairly constant with wire-drawn strain and at a level of elongation of 1% to 2%. Thus, at high strains, the wire from the zone-refined metal exhibits a higher room-temperature elongation, while at lower strains, its ductility is the same, or somewhat lower, than that of commercial beryllium. The reason for this is obscure at present.

The apparent decrease in ductility at the highest strain levels may be, again, the result of sampling error. On the other hand, this inflection may be the result of strain-induced recovery. If this be the case, then such recovery must be considered to embody grain growth and/or reorientation (partial destruction of the wrought preferred orientation) and might be described more precisely as strain-induced recrystallization at the working temperature.

It would appear to be necessary to repeat the work on the tensile properties in a more precisely controlled manner before drawing any really firm conclusions. The work in this area, which is reported above, should be considered to be quite primitive and to be indicative of some possible trends. The probability that these possible trends are valid is considered to be low at this time.

The swaged polycrystalline aggregates from crystal D were subjected to wire-drawing at 450°C. The drawing practice was identical to that used for samples 31 B and 31 C.

Sample D. 2a "broke up" continuously during wire drawing; approximately 1 inch of length was lost on each drawing pass by transverse cracking. These failures were fibrous in appearance. Three inches of stock remained after passing the 0. 0144-inch diameter die. This piece of stock was examined at 30X and was found to have both longitudinal and transverse cracks on its surface. Several places were noted along the longitudinal cracks where the defect was starting to spread so as to initiate a transverse defect. This was in accord with the opinion of the wire-drawing operator who felt that whatever was nucleating the transverse failures was originally within the wire and these nucleators were "working-out" to the wire surface continuously as deformation proceeded.

Sample D. 2b was drawn successfully to a final diameter of 0. 00407 inch. A few points were broken but no other failures occurred. No attempt was made to apply fresh lubricant at diameters below 0. 0075 inch (except to the newly pickled points). Sample D. 2b drew as well as or better than sample 31 C.

Specimens for tensile testing were cropped from each end of sample D. 2b at each of the following diameters*: 0. 03176 (1. 75),

*Parenthesized numbers indicate the wire-drawn natural strain as measured from the last anneal. Diameters are expressed in inches.

0.02712 (2.06), 0.02197 (2.48), 0.01688 (3.01), 0.01297 (3.54), 0.01051 (3.95), 0.00807 (4.50), 0.00654 (4.90), 0.00588 (5.11), 0.00530 (5.32), 0.00477 (5.54), and 0.00407 (5.85). These diameters were chosen so as to resolve some of the ambiquities of the plots in Figures 18 and 19. Unfortunately, funds were not available for this tensile testing, for an investigation of the failure of sample D. 2a, or for the reswaging and drawing of D. 2c.

Sample D. 2c and the tensile specimens from sample D. 2b were set aside for possible future work as was the scrap from sample D. 2a.

IV SUMMARY AND CONCLUSIONS

Zone-refined crystals of both thermally and electrolytically reduced beryllium were converted to polycrystalline aggregates by a combination of deformation and heat treatment. Deformation was accomplished at 450°C (generally) by both swaging and rod-drawing techniques. Swaging was found to be the more successful technique. The beryllium was jacketed in steel during these deformation procedures.

The resulting polycrystalline aggregates were subjected to deformation at 450°C by standard beryllium wire-drawing techniques.

Some of the zone-refined metal was shown to contain an appreciable quantity of non-metallic inclusions which were believed to be beryllium oxide. In some cases, the presence of these inclusions was believed to be the cause of catastrophic failure to deform by wire drawing. At least the material which did not fail during wire drawing did not show any evidence of a serious quantity of these inclusions. The inclusions, when present, did not seem to impede success during swaging but were judged to be detrimental during wire drawing.

Three blanks from two different zone-refined crystals (thermally reduced beryllium; one crystal was produced by The Brush Beryllium Company and the other crystal was produced by The Franklin Institute) were converted to fine wire at a reasonable material yield.

Some of this wire was tensile tested at room temperature. At high reductions (by wire drawing), the as-drawn wire approached the strength levels of commercially-pure wire. The ductility of the as-drawn wire was, in some cases, appreciably greater than that of commercially-pure wire in a comparable condition. Wire in the annealed condition was also tensile tested at room temperature. Strength levels were determined to be on the order of 50% of those of commercially-pure, block-pressed beryllium. The ductility of the annealed wire was found to be, in some cases, about 3 times that of commercially-pure, block-pressed beryllium.

A limited amount of metallurgical examination was accomplished by metallographic and X-ray diffraction techniques. Generally, the wire which was fabricated from zone-refined beryllium had the same metallurgical characteristics as commercially-pure wire. An exception to this generality was the response to annealing. It was found that the wire produced in this program exhibited a surprising capacity for grain growth, and there were indications that this wire suffered extensive reorientation during recrystallization annealing.

Some of the more important conclusions which were drawn from this program are:

1. The jacketed swaging technique can be employed to produce polycrystalline wire from single crystals of zone-refined beryllium.

2. The wire-drawing techniques which were developed for commercially-pure beryllium are satisfactory for the fabrication of fine wire of zone-refined purity. However, such zone-refined beryllium must be free from a serious amount of non-metallic inclusions.

3. The fabrication techniques which were used in this program did not cause significant chemical contamination of the zone-refined beryllium.

4. Two mechanisms must operate cooperatively in order to convert the single crystal into a polycrystalline aggregate. Part of the deformation must be a result of twinning, and some of these twinned orientations must either survive and/or grow during primary recrystallization to become grains in the recrystallized structure. Lacking either or both of these mechanisms, the wire will become more nearly an imperfect single crystal than a polycrystalline aggregate.

5. The two most prominent deformation mechanisms which are operative during swaging at 450°C are $\langle \bar{1}2\bar{1}0 \rangle$ slip and $\{10\bar{1}2\}$ twinning. The zone-refined metal behaved, in general, in a manner which was quite similar to that of commercially-pure beryllium.

6. Wire produced from zone-refined beryllium exhibits usable structural tensile-properties at room temperature.

7. Wire produced from zone-refined beryllium responds quite dramatically to recrystallization annealing with respect to grain boundary mobility and crystallographic reorientation.

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